# PETROLEUM REFINER

SPECIALIZED FOR THE OIL REFINING INDUSTRY, INCLUDING PETRO-CHEMICAL ENGINEERING, PETROLEUM SYNTHETICS AND NATURAL GASOLINE

March, 1946
Vol. 25, No. 3

*A Gulf Publishing Company Publication*
EDITED MONTHLY AT HOUSTON, TEXAS

GRADY TRIPLETT
*Editor*


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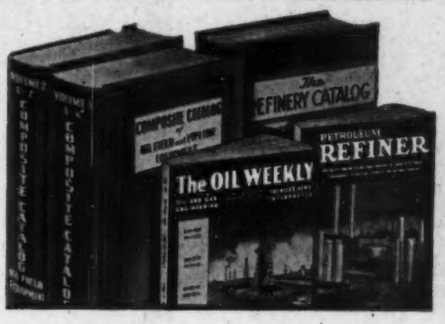


SPECIALIZED OIL JOURNALS


*Published by*
THE GULF PUBLISHING COMPANY
OFFICE OF PUBLICATION
3301 Buffalo Drive, Houston, Texas

R. L. DUDLEY, *President*
A. L. BURNS, *General Manager*
J. KENT RIDLEY, *Managing Editor*
TOM W. NELSON, *Advertising Manager*

NEW YORK: Room 523, 250 Park Avenue, Dick Swinsky, Manager.

CLEVELAND: 1010 Euclid Avenue, Robert B. Farson, Manager.

CHICAGO: 332 S. Michigan Avenue, H. G. Fitzpatrick, Manager.

LOS ANGELES: 426 W. W. Wilson Building, Huntington Park, California, J. W. Curts, Manager.

TULSA: 1304 Hunt Building, J. F. Carter, Jr., Manager

FT. WORTH: 203 W. T. Waggoner Building, H. H. King, Manager.

Single copies 35 cents (except special issues). Subscription price, domestic and foreign, $2.00 a year; 2 years $3.00; 3 years $4.00. Entered as second class matter June 16, 1923, at the post office at Houston, Texas, under act of March 3, 1879. Advertising rates on application.

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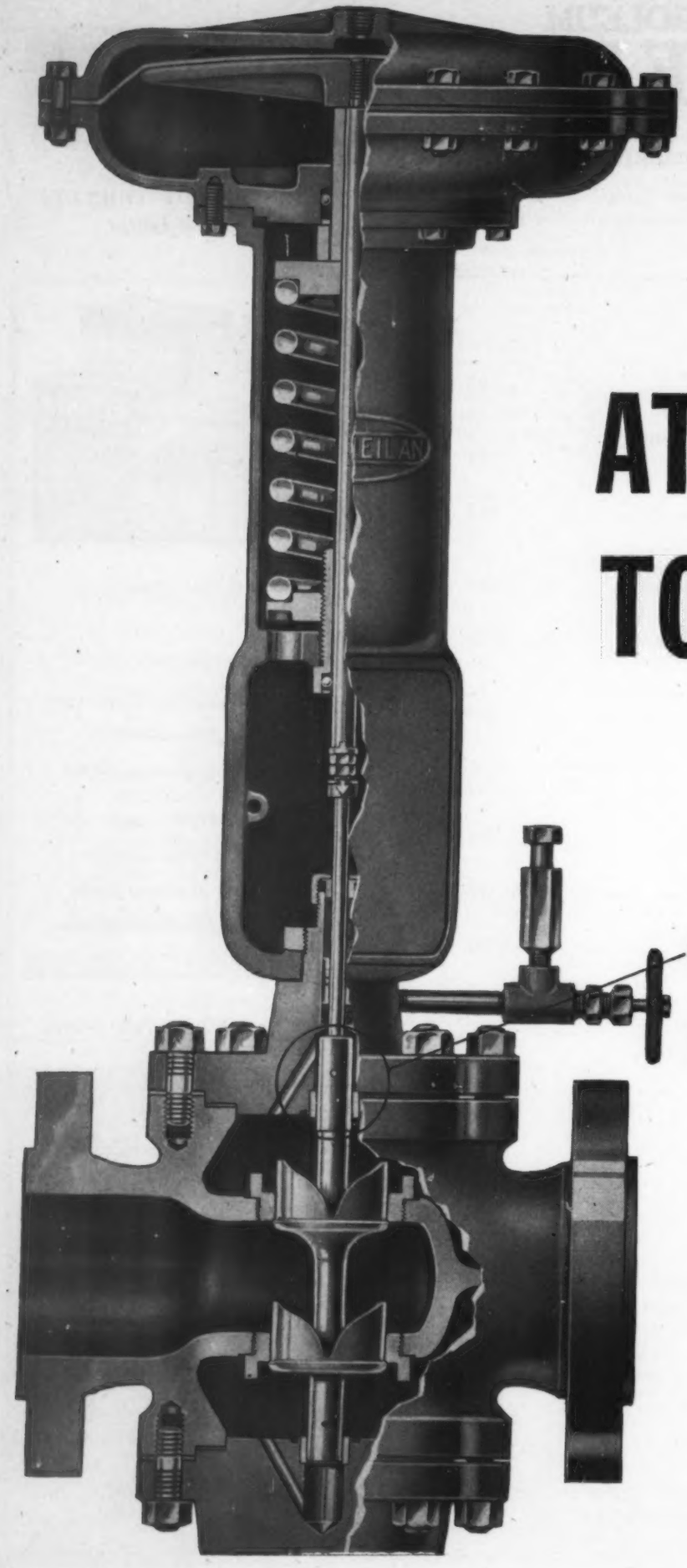


# ATTENTION TO DETAIL

## Pays Off in Control Valve Performance

Note: One skirt of the valve plug is 90° out of position in relation to the other.

VOLUME 25, NO. 3 MARCH, 1946

PETROLEUM
REFINER

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# THE LOOK BOX

AS REVISED, the bill to set up the National Science Foundation is an improvement over earlier efforts. For instance the report of the subcommittee on war mobilization has this explanation:

**Improved by Revision**

"In order that there be no confusion on the matter of patents . . . the proposed bill does not contain any provision for the modification of existing patent laws . . . "

While few supporters of our patent system contend that it is perfect, there does exist a determination to maintain a system wherein individuals and corporations may gain patents, hold them, transfer them or exploit them as desired. This attitude is based on the reasoning that in accomplishing a patent a man has created something of value, just as he has come into something of value when he builds a house or cultivates a crop. It is the simple conclusion that a patent is property.

Another worthwhile attitude of the senate subcommittee was expressed by Senator H. M. Kilgore, . . . "we are anxious to have further comments and constructive criticisms."

The danger in legislation for promoting research lies in the tendency to trust a few men with the responsibility of technical development. No set of men can be so trusted. Men are not competent to determine what projects deserve primary consideration. The nation will fare better if men are left to strive for patents according to their own estimates, however preposterous these estimates may seem. Dr. Willard H. Dow had a significant observation on that theme when he said:

"We have learned that if a research laboratory is to produce results, the men must be allowed the freedom to be a bit crazy. If we were to control closely, we would miss the great idea that is found only by those with the courage to be crazy."

Men in the refining industry, especially those engaged in research, will do well to let this committee know their attitude. They should let the law makers know that the privilege of searching belongs to the individual and that legislation should not hamper it. All too often legislators reveal the conviction that they are the keepers of privileges, with authority to dole it out to individuals. It is the other way around in a democracy. Privilege of research or any other privilege belongs to the individual. In setting up this government individuals granted to legislators the authority to regulate only a fraction of privilege. The constitution and its bill of rights are evidence of how small this fraction was measured.

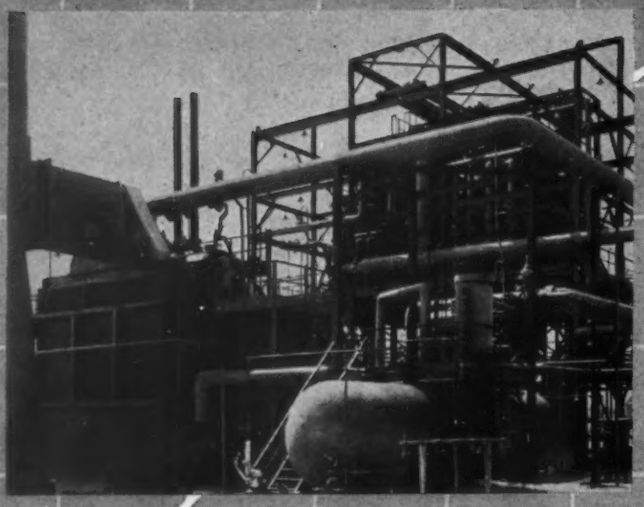
ABOVE the strife of industry there is an ideology of agreement. If this could be carried into the deliberations that attend the strife, idle plants would be few or none at all. Only active plants can fulfill the ideology of agreement.

**Strata of Agreement**

This level of agreement holds, for instance, to the conviction that children have a right to orange juice, warm clothing and schools. Or, it accepts the proposition that none should be hungry and poorly clothed. Only the willingness to work for the necessity is made a condition of its provision.

It is over the method of assuring orange juice, automobiles, radios, homes and schools strife

# Dowtherm on the job!



## Handling a big assignment in acid concentration

A battery of 30,000,000 Btu/hr. Dowtherm vaporizers supplies the heating requirements in this sulphuric acid concentration plant, built several years ago by a great Eastern firm. When the plant designers chose Dowtherm for this big installation, they simplified subsequent processing operations . . . and they also achieved a sizable reduction in the original construction costs of the plant!

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CHEMICALS INDISPENSAELE TO INDUSTRY AND AGRICULTURE

abounds. One group contends that profits be trimmed to provide more for the worker, who in turn will provide orange juice for his youngster. Another group contends that the road to plenty, whether it be orange juice of automobiles, is higher output per man or per machine. And the clash of methods results in shutting down the plant, which all agree must operate before men enjoy its output.

Somewhere there should be wisdom sufficient to lift the controversy into the strata of ideology. If men could cling to the knowledge that the American economy is capable of providing every industrious individual with necessities and luxuries, plants would be kept operating until men learned how to make a free economy serve them.

It does seem that an idle plant would convince all elements of its uselessness.

**Going Down**

CHEMICAL rubber is being produced for as little as 15 cents per pound. This accomplishment is in "our most efficient synthetic plants," to quote from a statement by P. W. Litchfield.

The lower cost recently was promoted by shifting to refinery gases for all butadiene, chief ingredient of chemical rubber.

The Litchfield analysis of the status of the chemical rubber industry, contains this significent statement:

"America could not long protect herself against aggression without an adequate supply of rubber. Without our synthetic plants, we would once more be dependent upon areas in the Far East for our rubber supplies. The Japanese taught us our lesson and we must never again permit ourselves to be caught in such a dangerous predicament. No matter whether the final solution is private or government ownership, America must keep intact adequate facilities for the production of rubber."

**Human Element**

IN THIS issue of PETROLEUM REFINER will be found the first part of a series of articles on elementary chemistry, prepared especially for men who work in refining without the benefit of school study of the subject. In large measure the articles have been selected so as to assist in a wider program of vocational training. Men, some with formal education, are back from military service and need a source of basic information. Also, the men who came into refining during the war need a similar source of information.

To the latter it is pertinent to point out that chemistry can not be made as simple as many desire. Study is essential to the learning of any science. Fortunately many plants have instituted courses for study and these articles may serve as text in chemistry.

Despite the requirement of study, it is more pertinent to point out that formal education is not essential to the learning of chemistry. The man who applies himself will grasp the fundamentals and find chemistry both interesting and valuable. The industry needs more men with more knowledge and it is fortunate that there is a program of human engineering, an effort to improve men along with the effort to improve equipment and processes.

**New and Different**

ADAPTATION of atomic energy to power production differs from its use in destruction as was explained by Dr. C. G. Suits before the American Institute of Electrical Engineers:

"We have during the war largely used up our accumulated surplus of basic knowledge of the atomic nucleus. The dividend was the atomic bomb. We must, without further delay, restore this surplus in preparation for the important peacetime job for the nucleus—power production.

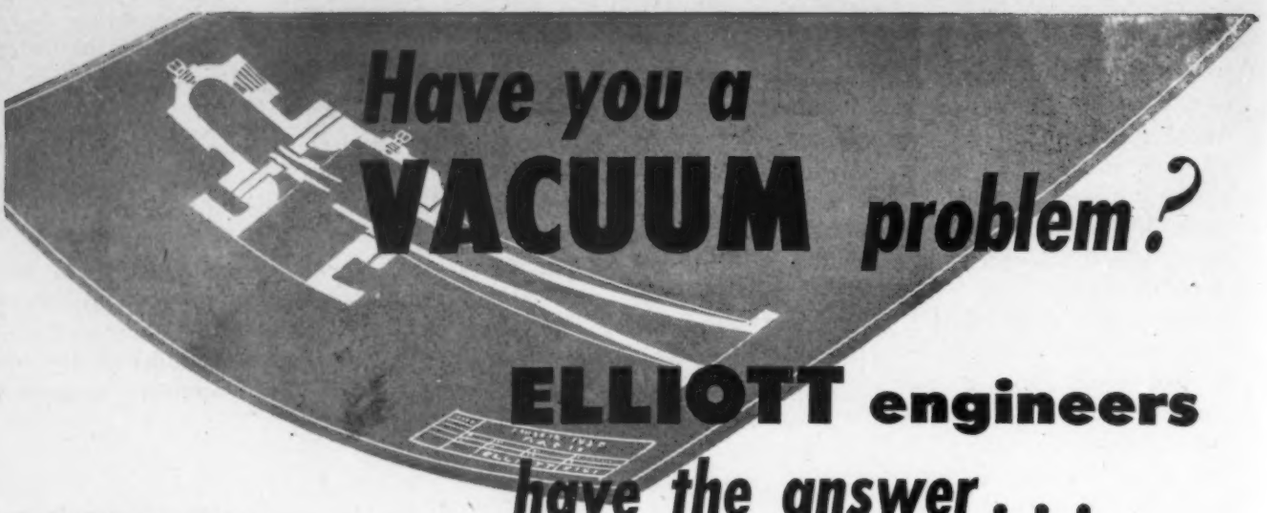
"The development of economical atomic power is not a simple extrapolation of knowledge gained during the bomb work. It is a new and different project and great effort will be required to reach a satisfactory answer . . . "

After making it clear that "chain reacting pile" of material containing uranium or other material together with moderators were required to produce the elements of the bomb, he noted that a minimum amount of 50 tons rules out many small uses such as the automobile, perhaps the locomotive. Of electric power plants he added:

"Atomic power plants for electric power generation will probably at some time in the distant future compete successfully with coal, oil and water energized plants. In fact this ultimate possibility is an important hedge against the ultimate exhaustion of the natural oil and coal reserves."

*"As we look at these problems of human relations, we feel that the solution must be found through a closer understanding between management and labor. If we cannot succeed by cooperation, it doesn't seem likely that we can succeed by any exercise of force. We cannot, for example, expect legislation to solve our problems. Laws which seek to force large groups of Americans to do what they believe is unfair and against their best interests are not likely to succeed. In fact, such legislation can lead to exaggeration of the very problem it is designed to solve. And when free men give up the task of trying to get along with each other, and pass the buck to government, they surrender a substantial measure of their freedom."*

—HENRY FORD II



# Have you a VACUUM problem?

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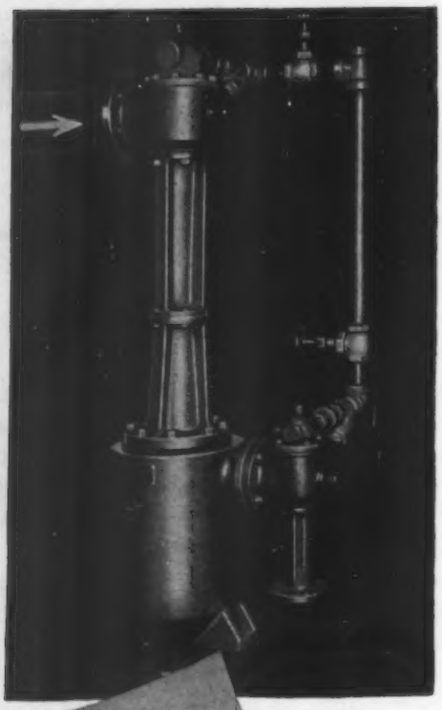

They have made a specialized study of the steam jet ejector over a period of 25 years, and were the first to go "all out" for the steam jet ejector as the modern vacuum pump. They developed the ejector to get constantly higher vacuum, working out multi-stage units for a wide variety of process applications.

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# Steam Jet Ejectors by ELLIOTT

G-392

# Corrosion in Hydrofluoric Acid Alkylation

F. A. PRANGE, Metallurgical Engineer, and R. A. FINDLAY, Chemical Engineer
Phillips Petroleum Company

PRIOR to the outbreak of war, petroleum engineers and operating men had had virtually no experience with handling of hydrofluoric acid. However, the tremendous wartime demands for aviation fuel necessitated rushing the HF alkylation process from a laboratory stage into large-scale production. No compromise could be allowed in considerations of safety in view of the possible hazards in using this chemical on a large scale and it was necessary to call upon the best metallurgical testing techniques for the selection of materials of construction for the necessary equipment. Before and during the period when Phillips Petroleum Company was designing the first HF alkylation unit ever to be built, a thorough, planned program was carried out which paid later dividends in continuity of operation and in an excellent safety record. The nature of this program is of interest, as well as the experience of nearly three years in large-scale use of hydrofluoric acid.

### Testing of Materials of Construction

Early experiments determining corrosion rates were carried out in the laboratory in bombs, both with and without agitation. However, limitations of this type of information were thoroughly realized and a comprehensive further study was planned and conducted to avoid any possibility of being misled by simple small-scale tests. Some of the more important factors which were considered are listed as follows:

(1) *Test Duration*—Short-time corrosion tests were recognized to be of limited value since scale on the metal surface is frequently the controlling factor as regards corrosion rate. Some metals (for example, aluminum and stainless steels) form a protective coating in the air. This layer may protect them for a short time in a new corrosive agent until the coating is completely penetrated. Other metals have no protective coating to start with, but form one after prolonged contact with a corrosive agent. In this case, a short test may be misleading in indicating too high a rate of corrosion. Testing varied from 6 to 40 days on a small scale and for as long as six months on a larger scale.

(2) *Bimetallic Corrosion*—To avoid accelerated corrosion caused by formation of couples between different metal samples, the use of Saran and Polystyrene insulators was practical at low temperatures. At higher temperatures, tests were made with acid dripping over the specimens. Other tests were run in which no effort was made to insulate the samples and the comparison of the two sets of results showed no significant bimetallic corrosion in substantially anhydrous acid.

(3) *Metallurgical Examination*—Any type of corrosion testing should be supplemented by careful metallurgical examination to detect types of corrosion other than simple metal loss. Several examples can be cited which arose during the investigations preparatory to commercialization of HF alkylation. Samples of Ni-Resist cast iron showed "graphitic" corrosion in concentrated HF, the iron being corroded and the graphite unattacked, leaving the sample in its original shape but so soft it could be cut with a knife. Intergranular corrosion was observed in Hastelloys A and B in the presence of aqueous HF, the grain boundary material corroding away, leaving individual grains to drop out.

(4) *Effect of Air*—The effect of oxygen from the air was noted early in the corrosion-testing program. In some cases a definitely increased corrosion rate was observed when the acid was pressured into the testing apparatus with air. This was of particular importance in the case of tests on Monel in aqueous acid where a type of corrosion known as "denickelization" was observed and was found to be due to the presence of oxygen. Tests involving accelerated corrosion caused by oxygen were not considered in choosing materials of construction since commercially-operating units would not contain appreciable amounts of oxygen during normal operation. Monel, for example, has proved a very valuable material of construction.

(5) *Effect of Velocity—The "Corrosion Pilot Plant."* The most important precaution which was taken in this testing program was the operation of a "corrosion pilot plant." This plant made possible the observation of rates of corrosion under conditions of high velocity and turbulence (such as in pump impellers) similar to those encountered in plant equipment. The "corrosion pilot plant" was merely a circulating system having a centrifugal pump taking suction from both the butane and acid phases of an accumulator and discharging back to the accumulator through a series of 2- to 4-inch valves, fittings, flanges, etc., through a case which contained corrosion test samples, through a commercial model flow controller, a water cooler or a steam heater. This simple circulating system simulated as much as possible the operation of actual equipment in HF-hydrocarbon service and made possible the testing of materials of construction and equipment under conditions very similar to those in the projected plant. Two valuable by-products of this method of corrosion testing were obtained. Various mechanical problems, such as operation and packing of pumps and valves, etc., were solved before the HF alkylation plant was

**TABLE 1**

**Average Rates of Corrosion in Substantially Anhydrous HF**

**(Inches Penetration per Year)**

| TEMEPRATURE (°F.) | 60-80 | 80-100 | 100-120 | 140-160 | 180-190 | 250-300 |
|---|---|---|---|---|---|---|
| Monel | 0.003 | 0.001 | 0.005 | ..... | ..... | ..... |
| Cupro-Nickel (30%) | 0.002 | ..... | ..... | ..... | 0.01 | ..... |
| Plain Carbon Steel* | 0.003 | 0.006 | 0.014 | 0.025 | 0.09 | 0.10 |
| Magnesium (Dowmentals E & J) | 0.005 | 0.017 | ..... | 0.000 | ..... | ..... |
| Antimonial Admiralty | 0.01 | 0.013 | ..... | ..... | 0.02 | ..... |
| Copper Alloys† | 0.02 | 0.02 | ..... | ..... | 0.06 | ..... |
| Aluminum | 0.02 | ..... | ..... | ..... | 0.98 | ..... |

* Low alloy steels (e. g. SAE 4140 and carbon-moly) have the same corrosion rates as plain carbon steel.

† High copper-content alloys such as pure copper, aluminum bronze, phosphor bronze, silicon bronze, and 444 bronze corrode at the same rate.

**TABLE 2**

**Rates of Corrosion in Aqueous HF (40-50% HF)**

**(Inches Penetration per Year)**

| TEMPERATURE (°F.) | 140-185 | 275-300 |
|---|---|---|
| Silver | ..... | 0.003 |
| Copper Alloys* | 0.003 | 0.02 |
| Monel | ..... | 0.03 |
| Magnesium (Dowmetals E & J) | 0.02 | ..... |

* Copper, red brass, antimonial admiralty, cupro-nickel, phosphor bronze, silicon bronze, and 444 bronze give similar corrosion rates.

constructed. Also the operating personnel were given a chance to become familiar with the handling of hydrofluoric acid. Furthermore, the "corrosion pilot plant" also contributed considerably to knowledge of corrosion rates. The main material of construction, carbon steel, corroded at a rate of only 0.003 inch penetration per year during 6 months' operation and was found satisfactory in valves, fittings, and even in the pump impeller. A good example of the advantage of this large scale investigation was the discovery made that 11-13 chrome stainless steel is sensitive to the velocity of flow. Although this steel had appeared to be better than carbon steel in small-scale tests, an 11-13 chrome steel pump impeller was very badly attacked after four months operation whereas carbon steel was unattacked after a longer period in the same service.

## Corrosion Rates

The corrosion rates of some of the more important metals as determined during this program are listed in Tables 1 and 2. The data for substantially anhydrous hydrofluoric acid (acid containing not more than 1-2 percent water) given in Table 1 indicate that carbon steel is a suitable material for equipment up to a temperature of about 150° F. Monel or Monel-clad steel and cupro-nickel are suitable for even higher temperatures. In aqueous HF (approaching the azeotropic mixture) copper and its alloys, and Monel show considerable resistance, and silver shows very high resistance. These data, plus the supplementary observations obtained during the investigation, were the basis for the design and selection of equipment for the first Phillips HF alkylation unit.

## Corrosion Experience in Plant Operation

One of the Phillips Petroleum Company units has by now operated nearly three years, and the other, somewhat over two years. Time on stream of 95-97 percent is a rough indication of the value of a preliminary investigation similar to that described. A general description of the HF alkylation process has been given previously[1] and need not be repeated here. In the commercial units, carbon steel was used for all equipment in the contacting section, where substantially anhydrous acid or acid-hydrocarbon mixtures are handled at about 100° F., and in the azeotropic tower, where substantially anhydrous HF-hydrocarbon mixture is fractionated overhead at about 150-170° F. Plain carbon steel was chosen as the main material of construction for the acid rerun section where hydrofluoric acid is purified by fractionation as a substantially anhydrous overhead product, oils and HF-water azeotropic mixture being drawn as a bottoms product. Monel linings were used in the lower section of this tower where the dilute acid and the highest temperatures are encountered. Column temperatures in the top of the acid rerun section are in the range 150-200° F. and bottom temperatures normally 275-300° F. Experimental bubble caps and plates of various materials have been tested in the commercial units to gather additional data on corrosion. The following notes summarize experience in commercial scale equipment.

*Carbon Steel*—Periodic inspection of vessels has shown corrosion rates of carbon steel equipment to be equivalent to or lower than expected. For the most part no change in thickness has been detected in vessel walls within the limits of error of this type of measurement, but it is realized that corrosion rates can be measured more accurately in experimental work. Additional evidence from examination of machining marks, undulation of the welds, and thickness of scale in commercial units all indicate a rate of corrosion below 0.01 inches per year. Figure 1 shows a tube bundle from an acid-hydrocarbon contactor. There is no apparent corrosion of this bundle which had been in service about a year and a half at temperatures in the neighborhood of 100° F. No measurable corrosion loss has been observed in azeotropic towers where acid-hydrocarbon mixtures are handled at 150-170° F. In the upper part of acid rerun columns where carbon steel is in contact with substantially anhydrous hydrofluoric acid at 150-200° F., thickness loss of cascade trays, bubble decks and caps has been in the range 0.03-0.05 inch per year although vessel walls have suffered much less attack in this location. Figure 2 shows some carbon-steel bubble-cap assemblies after a year and a half contact at temperatures around 175° F.

Experience with carbon steel in the bottom section of the acid rerun tower, where the product consists of an oil and HF-water mixture removed from the acid, has been varied. In some cases the corrosion was very considerably less than would be expected in dilute acid service and at temperatures from 275-300° F. However, the material being removed was an emulsion of aqueous acid and a heavy oil, and this may have retarded the corrosion by the HF-water mixture. Although Monel or Monel-clad steel is the recommended construction material for this service, a carbon-steel section was used for six months in one unit, at the end of which time it showed very little corrosion. In another unit a carbon-steel column was used experimentally for about 3 months in a different arrangement designed to remove the water separately from the oils, and corroded at a rate of 0.16 to 0.5 inch per year at different points.

*Monel*—Corrosion of Monel bubble caps, chimneys, trays, and linings in the acid rerun tower at 150-200° F. in substantially anhydrous acid has been very slight (see Figure 2). This is the most turbulent and severe condition under which concentrated acid is used in the unit and corresponds to the point where the highest loss has been observed for carbon steel. No metal loss has been measured in Monel equipment

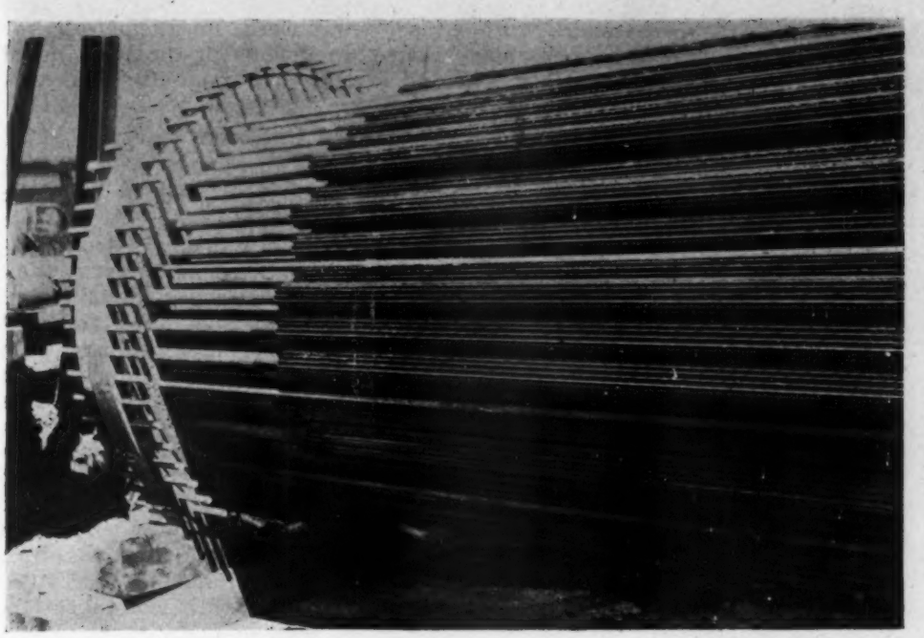

**FIGURE 1**
**Tube bundle after 18 months service (in substantially anhydrous HF-hydrocarbon mixtures at about 100°F.)**

under these conditions, indicating a very high resistance.

In aqueous acid service, Monel has been used as a lining material in the lower part of the acid rerun column. No appreciable corrosion of the Monel has been observed. The presence of the aqueous acid in an emulsion with heavy oil may, however, have decreased the corrosion rate below a normal value for true aqueous acid service.

*Copper Alloys*—Observations on pilot-plant equipment had indicated that various copper alloys corroded more rapidly in high-velocity streams of concentrated hydrofluoric acid. Test bubble caps of Everdur (silicon bronze) and copper and one Everdur tray were installed in acid rerun towers to check these materials. Corrosion under this degree of turbudence amounted to about 0.12 inch per year as compared with 0.06 inch per year in the smaller scale tests listed in Table 1. In eight months operation in one unit, these caps and the tray were almost completely destroyed. The rate of corrosion of copper was somewhat higher than that of silicon bronze.

*Antimonial Admiralty Metal*—A heat exchanger was installed in one unit with antimonial admiralty tubes because it was available at the time. After only two months acid service at about 190° F., the tubing was badly pitted. This may be compared with the resistance of seamless steel tubes which showed no measureable loss on the acid side of the tube after a year and a half service. Antimonial admiralty is similar to other copper alloys in being sensitive to velocity of flow.

*Cupro-Nickel*—Although no plant experience is available with cupro-nickel, laboratory experience has indicated that its corrosion rate is not affected by velocity and that it is a suitable material of construction up to 200° F. for concentrated acid service. Since it also has good resistance to aqueous acid, cupro-nickel might prove satisfactory for equipment where acid strength varies considerably.

*Stainless Steels*—The "corrosion pilot plant" had warned that 12-chrome stainless corroded rapidly in high-velocity streams of anhydrous acid and this material was generally avoided in plant construction. A few gate and motor valves were installed with

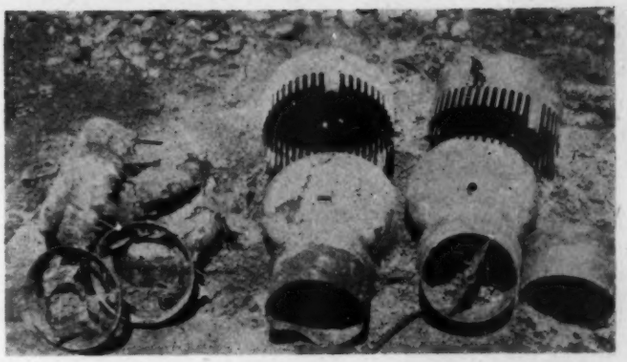

**FIGURE 2**
**Bubble cap assemblies after 18 months service (in substantially anhydrous HF at about 175°F.). Carbon steel at left, and monel at right.**

12-chrome discs and were found to undergo rapid corrosion and erosion. Some 18-8 stainless steel has been used in commercial units for thermocouple wells, orifices, and trim for relief valves and diaphragm motor valves. This steel has given fairly satisfactory service although it has not shown appreciably higher resistance than ordinary carbon steel. Even at high temperatures in the bottom of the acid rerun tower, 18-8 thermocouple wells do not have longer life than carbon steel ones. In view of comparative cost, stainless steel is not given much consideration for HF service.

*Silver*—Silver was used very little as a material of construction and then only where there was no chance of excessive wear or erosion, for example, in plating relief valve springs.

## Fabrication Techniques

The possible influence of construction techniques on corrosion received attention during the corrosion investigation and later experience has confirmed the decisions made at that time.

*Stress-Relieving*—Early experiments indicated that carbon steel showed no signs of stress corrosion in concentrated acid. In building commercial-scale units, while pressure vessels for acid service were stress-relieved, none of the field welds in the piping or vessels were specially treated. In all of the equip-

**FIGURE 3**
**Monel lining—lower section of acid re-run tower. (Note distortion of lining due to corrosion of shell underneath.)**

ment in use no evidence of stress corrosion has appeared after well over two years of operation.

*Steel Castings*—Either cast or cold-rolled carbon steel offer equally good resistance to concentrated hydrofluoric acid. However, castings must be sound for HF service, since small sand inclusions or slag pockets will be immediately attacked by the acid, causing leaks.

*Welding*—Care must be exercised in welding to obtain slag-free joints since any slag pockets are rapidly attacked.

*Brazing*—Silver solder for use in high-velocity streams of concentrated HF should be of high-silver content. ASTM Grade No. 7 silver brazing alloy has proved satisfactory for severe service in laboratory work.

*Monel Linings*—Welded strip linings of Monel have been used but have shown a tendency for leaks to develop in the welds. In more concentrated acid service this has not caused any difficulty but in aqueous acid service in the lower section of the acid rerun tower, accelerated corrosion of the steel tower under the lining has been observed (see Figures 3 and 4). Monel-clad steel is recommended where practical.

*Copper Welds*—Welds in deoxidized copper plate made with copper rod were unsatisfactory for aqueous acid service. Inclusions in the weld metal were attacked and in less than a month, the welds became porous.

## Localized Corrosion

As stated, carbon steel is a satisfactory material of construction for use in concentrated hydrofluoric acid service. However, very rapid failure is possible under certain conditions if a dilute acid phase is permitted to collect at some point during the process of vaporizing or condensing this acid. Examples of failures taking place in about one month in steel tubes of heat exchangers and condensers have been cited in a previous publication.[2] It is important in design of carbon-steel equipment to avoid dead spots where dilute acid can accumulate during vaporization by utilization of fast-moving, turbulent flow which will result in maintaining mixing. In the case of water coolers and condensers, construction should be such that very small leaks in gaskets or tube rolls can not be present since trace leaks result in the formation of pockets of dilute acid on the water side, causing failure of the equipment. Welding of tubes into tube-sheets has been adopted in many cases to prevent traces of leakage.

Special precautions were taken in design so that all equipment could be completely drained during periods off stream to eliminate the possibility of formation of pockets of dilute acid.

## Conclusion

Thorough metallurgical investigation and the use of a "corrosion pilot plant" did much to facilitate the rapid development of the HF alkylation process from a laboratory scale to a plant scale. Ample large-scale experience, combined with these investigations, has demonstrated that carbon steel suffers very little corrosion in equipment in this process up to temperatures of about 150° F. Monel is satisfactory for considerably higher temperatures and is adequate for the severe conditions of aqueous acid plus oils drawn from the bottom of the acid rerun unit at 275-300° F.

Copper alloys, with the exception of cupro-nickel have been found unsatisfactory for concentrated acid service where liquid velocities are high. Chrome stainless steels are also sensitive to velocity.


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**FIGURE 4**
**Lower section of acid re-run tower—external view. (Ruptured due to accelerated corrosion under lining.)**

# Treatment of Hot Resin Burns and

# Safe Handling of Thermoplastic Materials

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**THIS discussion of burns resulting from accidental contact with hot asphalt-like materials is presented primarily from the first-aid viewpoint, although one important feature of the subsequent treatment is suggested for consideration by the medical profession. The methods herein proposed involve two radical departures from ordinary procedures. The first-aid steps include:**

**1. Cool rapidly with water.**

**2. Treat for shock.**

**3. Call a doctor.**

**4. Apply a burn ointment or medicated jelly over the resin and the surrounding areas, and bandage loosely.**

**5. Do not remove the resin.**

**In addition to the actual burn treatment, some definite recommendations are made regarding the handling of hot resinous or thermoplastic-type materials, both in the plant and in the laboratory. The desirability of more effective cooperation between the chemists working with asphalts, resins, plastics, etc., and the doctors and hospitals serving these industries is also stressed.**

IN SPITE of the wide application, industrial importance and ever-increasing use of thermoplastic materials having asphalt-like physical properties, there is a literal dearth of literature on the safety factors involved in their handling or on the treatment of burns resulting from accidental contact with the hot molten materials. Still there are many "tricks" and techniques, that can be derived from the experience of the older asphalt industry, which should be disseminated and discussed throughout all industries, plants, and laboratories working with hot, sticky, thermoplastics, resinous substances. The object of this paper is to present some of these experiences on the safety factors and to suggest a new treatment for hot-asphalt burns.

### Type of Burn Under Discussion

Resinous and thermoplastic materials are normally handled at high temperatures. For asphalts, these temperatures frequently range from 650° to 850°F. in the refining processes, from 475° to 525° F. in the blowing stills, and from 300° to 500°F. in blending tanks and transfer lines. In addition, many of the laboratory tests involve heating the product to elevated temperatures. In the course of handling these materials—loading, pumping, blending, sampling and testing—there is ample opportunity, as most asphalt men will attest from bitter experience, for some nasty burns.

Contact is usually the splattering, blotchy type resulting from faulty valves, broken flanges, spilling, dropping, etc. The hands, arms, face and neck are the parts most commonly burned and the area affected may vary from extensive portions down to a few blotches the size of a dime. Because of the high specific heat and the sticky, plastic nature of these materials, the heat is retained and results in deep third-degree burns which are normally slow to heal and often leave unsightly scars. Thus the type of burn under discussion herein is that resulting from accidental contact with hot, sticky, high-specific-heat material which would normally cause a third-degree burn.

This discussion applies equally well to a variety of materials, of which asphalt is probably the best known and most widely used. Many of the resins, both natural and synthetic, some of the plastics and rubber-like materials, most waxes, tars and pitches, the extraction residues from the refining of lubricating oils, and other similar materials which are finding their way into industrial applications in ever increasing amounts, come under this category. As used herein, therefore, the term "resin" will be used in a very broad sense to designate all thermoplastic materials having asphalt-like physical properties, but which in themselves are not harmful to the skin at room temperatures.

### First Aid Measures

The first-aid measures for burns of the type described above are given and discussed below as a series of steps. It is generally accepted by doctors and first-aid authorities alike that the two principal dangers of all burns are infection and shock. The following treatment is designed to minimize both of these dangers for the particular type of burn under discussion.

STEP 1. *Cool rapidly* by immediately dipping the burned area in water or by flushing copiously with water. The primary object of this part of the treatment is to minimize the third degree burning by conducting the heat away from the hot resin as rapidly as possible.

Sometimes there is reluctance to put a burned member of the body under water, probably inspired by the belief that "water will cause blisters." As a result the patient who has a hand splattered with hot resinous material at a temperature of 500°F. or

higher will stand and wave his hand in the air while the sticky masses of resin retain the heat and burn deeper into the underlying tissues. Air is a relatively poor heat-transfer medium and as the resin cools, it skins over and solidifies on the outside, which further impedes the heat transfer. Some blistering is inevitable anyway and the patient is lucky if the burn can be held to the second-degree stage.

### Dissipation of Heat

The concept that water will "drive the heat in" is not correct, inasmuch as heat moves away from hot to cold and the greater the temperature gradient the faster the heat transfer in the direction given. The water merely provides a more rapid dissipation of the heat away from the hot areas.

The conservation of body heat is vital in minimizing shock. Therefore we recommend that only the part or member burned, such as the hand or arm involved, be immersed in water. Further conservation of body heat takes place in the later handling of the patient by the application as soon as feasible of blankets and hot water bottles to the body and extremeties. The relief of pain, until opiates can be administered by a doctor, resulting from the cooling water will also tend to minimize shock.

Thus, as an immediate Step No. 1, we recommend that the burning heat be removed from the hot mass of adhering asphalt-like material as rapidly as possible by immersing in cold water. We realize that this new concept is a deviation from some accepted beliefs, but, with the admitted deficiencies in first-aid treatment in these types of burns, we are convinced that it is a good and safe procedure.

In one case brought to the writers' attention a glass laboratory blowing still exploded in a chemist's face splattering arms, neck and face with blown asphalt at 500°F. He immediately stepped to the sink and splashed tap water in his cupped hands on to his face. Today his face which received the brunt of the hot asphalt is not scarred except slightly around the hair line while his upper arms which did not get the cooling water soon enough are quite badly scarred.

### Treat for Shock

Step 2. *Treat for shock.* Here one follows the straightforward Red Cross First-Aid shock treatment of "Heat—Position—Stimulant." Assist the patient to a recumbent position, preferably with the head lower than the feet, and keep him warm with blankets or other suitable means. Reassure the patient and do all possible to allay his fears. A stimulant may be administered: ½ teaspoon aromatic spirits of ammonia in ½ glass of water is the best first-aid stimulant, although hot coffee or tea taken in sips are also suitable. Whiskey is not a good stimulant because of variation in individual reactions and its subsequent depressant effects.

All burns are accompanied by some degree of shock. In many cases the subsequent treatment by the doctor is hampered or must be postponed because of shock. No matter how slight the shock may seem at first, it should be treated because it is strongly cumulative and the standard shock treatment is also shock prevention.

Step 3. *Call the doctor.* This is best done by a third person as soon as possible after the accident. In summoning professional help, be prepared to give explicit and complete information, covering the following points:

(a) Location of injured person.

(b) Nature, cause and probable extent of injury and the supplies available at the scene of the accident.

(c) What first-aid is being given.

Doctors are not mind readers and often, in the excitement following an accident, essential information is omitted or inadequately transmitted. If the patient is to be taken to a hospital or plant infirmary or first-aid center, a telephone call is in order so that preparations can be made to receive the patient to best advantage.

Step 4. *The first-aid treatment* of the burn itself is very simple. At the same time that the patient is being treated for shock, any one of the recommended burn ointments or medicated jellies is applied *over the asphalt* and surrounding burn areas. Ordinary mineral oil is both soothing and a fairly good softening agent. Other good agents are gentian violet or merthiolate jellies or ointments. Amertan, Tannipaste, and Tannafax are good. The burn is then bandaged loosely.

Step 5. *Do not remove the resin.* This is recommended without reservation as a first-aid measure and is discussed as part of the subsequent treatment of the burn in a later section of this paper. There is absolutely nothing to be gained by immediately attempting to remove the resin. On the other hand waiting does have some definite advantages and may prevent much serious damage.

### Hot Resin Sterile

Leaving the resin on provides the most effective first-aid treatment to guard against both infection and shock, the two principle dangers of all burns. In second- and third-degree burns plasma or blood serum loss is minimized. Shock is quickly precipitated by loss of this valuable fluid. In severely burned cases, plasma transfusions are needed when this serum loss is great.

Resinous materials at 300° F. or higher are sterile and so form a good protective covering over the damaged tissue. Removing the resin exposes the whole area to the ingress of infection and there is always danger of further damaging the underlying tissues by lifting pieces of the burned and weakened flesh with the adhering resin. The simplest and gentlest handling is vital. Even under the best of conditions removing the sticky, hard, asphalt-like material from a sensitive area is a slow and painful procedure. This is best done later when the patient has received an opiate or anesthetic under a physician's care. Obviously the danger of the patient going into severe shock is materially reduced if this ordeal is postponed at least until the initial shock has subsided. Thus, as a first-aid measure, leaving the resin on minimizes shock, protects against the ingress of infection and helps retain blood plasma.

The burn ointment and sterile dressings are applied as treatment for the areas immediately adjacent to the resin spots which are usually burned slightly (first degree). If the burn ointment should be a type that is miscible with the resin, the ointment softens the resin and thus makes its subsequent removal easier. The bandage helps protect the burned areas from accidental jogging or improper handling. Also, the application of any dressing has a psychological reassuring effect on the patient in making him feel that his burns are being treated and cared for.

These then are the recommended first-aid measures to be taken in case of burns resulting from accidental

contact with hot-sticky, asphalt-like materials which would normally cause third-degree burns. They include:

1. Cool immediately with water.
2. Treat for shock.
3. Call a doctor.
4. Apply a burn ointment or medicated jelly *over the resin* and the surrounding areas, and bandage loosely.
5. *Do not remove the resin.*

### Subsequent Treatment of the Burn

It is suggested that as part of the subsequent treatment of the particular type of burn under discussion, it might be advisable to not remove the resin for a matter of days. The whole severely burned area is sterile and the resin forms a good impervious protective coating which allows the damaged tissue to heal from the inside out, while effectively preventing the ingress of infection. As discussed below, the resin can only be safely removed with solvents and this is best done after the skin has had a chance to toughen a bit.

In general, pure petroleum asphalts are perfectly bland and have no harmful or irritating effect on the skin at room temperatures. Just how far one can go with other asphalt-like materials such as the resins, plastics, etc., will depend on their particular nature and history. Certain resinous materials, for instance, are extracted from petroleum in the refining of lubricating oils and, while the resin itself may be harmless, there could be enough of the extracting chemical, such as phenol, left in the resin to make it irritating to the skin. On the other hand, propane-precipitated resins are in general harmless like pure asphalts. Herein lies the first opportunity for cooperation between the chemist who is familiar with the nature and history of the resinous material and the doctor who must make decisions on just how the burn is to be treated.

The writers know of just one case where the resin has been left on a real third-degree burn for longer than a few hours. The thumb and index finger of an engineer were burned through accidental contact with a condensed petroleum resin at 500°F. Essentially the treatment as given in this paper was followed. The resin was not removed until nearly three days after the accident and then it was removed primarily because a grease-base burn ointment had been used which had softened the resin making it rather messy. The damaged area under the resin had started to heal nicely and the new skin could "take" the cold solvents used to remove the resin without causing pain or irritation. The new skin on the thumb was accidentally broken open the next day which slowed its progress slightly, but the overall recovery from the burn was considered excellent.

### Removing the Resin

The resin is best removed with suitable solvents. Scrubbing with tincture of green soap or washing with alcohol is futile. Vigorous scrubbing with soaps or irritating solvents only injures healthy tissue. Authors on the treatment of burns admit the inadequateness of most cleansing methods used at present. As previously mentioned, certain types of burn ointments will soften the resin and facilitate its subsequent removal.

If possible, as in the case of a hand or arm, it is desirable to use a large basin and enough solvent so that the whole member can be immersed. In any case dissolving the resin is usually a slow, tedious process and there is a tendency to become impatient and pick at the resin. This is undesirable as there is always danger of further damaging the underlying tissue. The solvent itself should be slowly agitated and gently washed over the resin with sterile soft-cotton swabs. *Don't be in a hurry!*

The most suitable solvent for removing the various resinous materials which might cause this type of burn will depend on their particular chemical structure and history. Recommended solvents for pure petroleum asphalts are high-flash naphtha, kerosine or gasoil. Benzol, gasoline, carbon disulfide, carbon tetrachloride, trichloroethylene and many of the other good solvents are either dangerously inflammable or irritating to the skin or both.

Some resins and plastics are soluble in alcohol but practically unaffected by petroleum solvents. Others may be best dissolved in ketones or esters or some mixed solvent. The doctor or nurse attending the patient cannot be expected to know the most suitable solvent for each of the many synthetic or natural resinous materials, even if they could recognize the particular one or the particular blend of such materials with which they were currently affronted. The chemist who actually supervises that operation or works with resinous material is the person best able to supply the desired information.

### Solvents for Hospitals

Herein lies a second opportunity for cooperation between the chemist and the doctor treating such burns. This cooperation will be most effective as foresight rather than hindsight. It is recommended that the chemist in charge of the particular project or the safety engineer, in case of plant operations, ascertain and supply to the hospital well-labelled bottles of the most suitable solvent for each asphalt-like material used in that plant or laboratory. Further the company doctors should be kept well informed by letter about the new resins or plastics and their peculiarities as each comes along and is experimented with or adopted. Some *one* should be assigned the responsibility of seeing to it that such information is properly disseminated, that the solvent bottles are kept full, and that the system does not bog down after the first burst of enthusiasm. This is not such an arduous task as it may seem at first thought because many of these resins can be classified into definite types with a solvent for each type.

It should be remembered that the best solvent for any given material may not be the most suitable for this particular use. In general, aromatic and chlorinated solvents tend to be irritating to the skin and these solvents will come into intimate contact with sensitive, burned tissues. The flash and fire point should be high enough (100°F. or above) to remove the solvent from the dangerously inflammable category. It has been found too that powerful penetrating detergents such as sulfonated petroleum hydrocarbons when used in conjunction with a suitable solvent are valuable. After the resin has been dissolved, sterile running water is best to flush the burned area and readily washes off the detergent and emulsified solvent mixture. Carefully following the above procedures leaves a surface ready for application of tanning agents or dye treatments. These subsequent treatments, of course, are carried out by a physician or under his supervision. The cleansing procedure too is often carried out under an anesthetic or after giving opiates.

### Some Points on the Safe Handling of Thermoplastic Materials

In keeping with the first part of the proposed treatment for resin burns, it is recommended that open barrels or pails of water be provided at the various places where accidental contact with the hot resin is most likely to occur. Such places might

include (1) the hot-oil pump room, (2) adjacent to sample lines on the stills or reactors, (3) points at which drums or particularly cartons are filled with the hot material, (4) points on roofing, laminating or coating machines where samples are taken or screens must be kept open or baths of the hot resin stirred by hand. These water barrels should be painted a distinguishing color and, with a little thought, can be so placed that they do not interfere with normal operations and still be handy in case of accident. A thin film of lube oil on top of the water will inhibit evaporation and prevent mosquito breeding. Some *one* person should have the responsibility of filling the barrels periodically. In the laboratory running tap water is usually readily available at the sinks. A shallow container large enough to immerse a hand may be kept in the sink and the tap water left running slowly during working hours as an added precaution. This provides cold water at all times and makes a convenient place to cool softening point rings and other small samples.

Contamination of the burn from these procedures is largely prevented by the overlying resin. Agents commonly used now in the subsequent medical treatment of burns include the powerful new sulfa drugs, administered both locally and internally. Penicillin too is being studied for use in treating the burn and any possible infection. In view of these effective anti-bacterial measures, the contact of unsterile water with the reddened skin adjacent to the resin is not dangerous.

### Protection for Workers

When working with hot resins, the sleeves should be rolled down to help protect the arms. Gloves with gauntlets that come well up over the wrists should be provided. An experienced asphalt man usually picks gloves that are a little too big so that they can be removed with a flick of the wrist, particularly after they get a bit dirty and sticky on the outside. The writer prefers plain double-thickness cloth or canvas gloves with a stiff gauntlet. These are relatively cheap and, therefore, can be replaced more often. Leather shrinks and shrivels the first time one touches a hot steam line or sample can, and asbestos gloves retain the heat and are too friable for general-duty plant use.

The use of a glove with a gauntlet has one serious disadvantage in that the hot resin may run down inside the gauntlet causing a more severe burn than if the wrists had been left bare. Suitable gloves with high tight-fitting wrist bands are unsatisfactory because they are too hard to put on and take off. The writer is not aware of a fully satisfactory answer to this gauntlet question for general resin work. Fortunately the type of accident wherein a stream of hot resin could run into the glove gauntlet is not common.

For the obviously hazardous operations, such as filling drums or cartons directly from a hot still, there are a number of special protective devices available.

There are a number of good face shields on the market that are quite comfortable to wear, allow full vision, and adequately protect the whole face from splattering liquids, etc.

In the laboratory it has been the writers' experience that faulty tongs or tongs not designed or meant for resin work are the cause of many accidental burns. Ointment cans or tins usually are found to be more convenient than glass beakers or flasks for much of the laboratory work on asphalt-like materials. Ordinary crucible tongs are not suitable for handing these tin cans, particularly the 8- or 16-ounce can sizes. What happens with the ordinary long-jaw, narrow-tip tongs is that the tips slip by one another over the thin metal giving the can a sudden side ways jerk which, frequently splashes the hot contents onto the operator's hands.

### Eternal Vigilance

In the plant, proper equipment, adequate instruction and eternal vigilance are the watch words for the safe and efficient handling of thermoplastic materials. Every valve on a line should be checked before a stream is pumped down that line. Steam-jacketed or steam-cored lines, particularly those that have not been used for some time, should be thoroughly checked to be sure there has been no steam leak into the line; also, the piping in steam-heated tanks should be inspected periodically. It takes only a small amount of water to raise havoc when brought in contact with hot viscous materials.

Valves in asphalt service have a tendency to "freeze" and it is sometimes difficult to tell whether a valve is open or closed. Rising stem valves, of course, tell at a glance the position of the gate but the safest way is never to leave a valve "full-open." If a valve is opened full and then "backed-off" about one quarter to one half a turn, one can then tell immediately that the valve is open by "feel." This is a good general rule to follow with all valves, regardless of the service. Wheel wrenches are handy tools but should be used with caution. The writer has seen the stems of valves twisted completely off by using too much force with a wheel wrench. Warming a valve with a little open steam will often save time and accomplish more than brute force in thermoplastic resin work.

Some additional miscellaneous points on the safe handling of resins might include:

1. The handles of sample buckets should be at least two feet long and should be rigidly welded to the bucket. Wired handles or ordinary pail type sample buckets are dangerous.
2. Clear lines well after use. This is best accomplished by blowing back with compressed air.
3. Avoid "shutting-off" against a pump, particularly reciprocating types.
4. Never work on a line or valve that is under pressure.
5. Thoroughly instruct new men and give them adequate time and training under an experienced operator with a good safety record.
6. Arrange periodic open discussions to review and refresh memories for all concerned on safe methods, operating techniques, etc.
7. Make complete accident reports and see that they are circulated or posted so that everyone will have an opportunity to profit by the experience and avoid repetition of the accident.

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# Practical Refrigeration for Natural Gasoline Plants

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THE advantages of refrigeration in natural-gasoline-absorption-plant operations do not seem to be as generally appreciated as is justified. We are at a loss to understand the reason for this since refrigeration units have been used in a few natural-gasoline plants for a number of years. The literature is also reasonably complete on the theoretical design[1] and advantages[2] of a modern unit. However, it does appear that a more detailed study of the practical design, operating problems and actual data may serve to emphasize the value of refrigeration in a modern low-pressure absorption plant.

In California there now seems to be a very definite interest developing in this type of unit. Possibly this is the normal reaction that may be expected as a result of the installation of at least three refrigeration units in existing low-pressure absorption plants during the last year and the inclusion of such a unit in a new plant that is now being constructed. These existing refrigeration units have all proved extremely economical to install and are efficient and simple to operate. It is therefore natural that the interest in this type of operation will grow as this information is appreciated. For the same reason additional installtaions of refrigeration units should be expected in the near future. The writers have been directly interested in two of these recent refrigeration plant installations and their actual design and operation is the real basis of this paper.

We will limit our consideration to the use of propane as a refrigerant and to the application in low pressure absorption plants with absorber pressures of less than 100 psi. Propane has the advantage of being inexpensive, readily available and of high refrigerating efficiency. Operating pressures are above a vacuum which precludes air contamination and all pressures allow reasonable pipe-line sizes without serious pressure drops.

OIL TEMPERATURE VS. EQUIVALENT OIL RATE

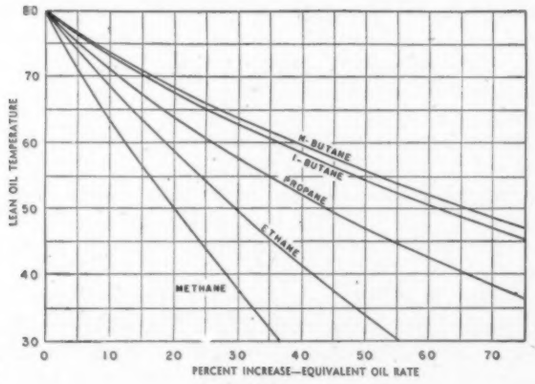



**FIGURE 1**

**Data developed at 55 pounds absolute and for an 80° F. base. Spread in effective vapor pressures can be shown as reciprocals of these values.**

**REFRIGERATION can be justified in a new low-pressure absorption plant and in cases where the future maximum volumes of wet gas are known within reasonably close limits, or where the atmospheric temperatures and humidity are too high to allow efficient atmospheric cooling. Refrigeration is also justified in many high-pressure operations but this paper is limited to low-pressure absorption where the advantages to be gained are probably more pronounced due primarily to the fact that a greater cooling range is possible without danger of hydrate or ice formation.**

**Refrigeration is also felt to be the simplest, cheapest and most economical means of securing additional plant capacity in existing plants. Experience has shown that the problems of operating and maintaining a refrigeration plant are extremely few and of minor importance. It also indicates that no unusual hazards result from the use of propane refrigeration. It is believed that practically any plant can develop a supply of propane at very small cost that will prove acceptable as a refrigerant for this type of operation.**

**Although papers have been presented in the past dealing with the advantages of propane refrigeration, their value apparently has not been fully appreciated. It is hoped that the presentation of this information on a practical rather than a theoretical basis will prove the key necessary to opening the door to a more general acceptance of this type of installation, which the authors feel to be justified on a practical and economical basis.**

**This paper was presented before the California Natural Gasoline Association, Los Angeles, January 3, 1946.**

## Spread of Vapor Pressures

The advantages of refrigeration are not alone due to the reduction in vapor pressures but also the fact that the heavier fractions are reduced to a relatively greater extent than are the lighter hydrocarbons below the butanes. As a result there is a greater spread of vapor pressures between the various hydrocarbons at the lower temperatures. This results in better separation of the fixed gases from the fat-oil tank and wherever colder temperatures are developed. An example of this condition is shown by Figure 1, showing oil temperatures against increased absorption below 80° F. in terms of increased oil rate to secure equivalent results. From this curve it

can be found, for example, that by lowering the oil temperature from 80° to 55° F. that this is equivalent to a 50 percent increase in oil rate at 80° F. insofar as the butanes are concerned but only 35 percent for propane, 24 percent for ethane and 17 percent for methane. This advantage of selective absorption is not available by the other means of increasing plant capacity such as increased oil rate, lighter mol-weight oil or increased pressures.

The costs of installing a refrigeration unit capable of subcooling the lean absorption oil by 25° F. are small interms of equivalent oil rate and it would appear that such a unit might easily be justified in a new plant. This would be especially true where the volume of gas to be treated may be expected to remain relatively constant and the prospects of future additions to the plant to be unlikely. It is also true where poor conditions of atmospheric cooling are obtained which would require an oil circulation that could not be justified when compared with the corresponding costs of using a lower oil circulation and installing propane refrigeration to secure the same results. Where future expansion seems likely, it would appear reasonable to design the plant with the thought that refrigeration might be added later.

### Savings in Costs

Benefits of refrigeration are more easily appreciated in existing plants where it is necessary to increase the plant capacity for the purpose of extracting additional light hydrocarbons or to regain a plant efficiency that has been reduced due to additional load. Under these conditions the costs of installing the additional equipment for the necessary oil circulation (and additional plant units) can usually be shown to be much greater than the corresponding cost of installing a refrigeration unit to accomplish the same results. In addition the actual installation of a refrigeration unit is a simple problem and can be accomplished without the usual interference with the existing plant units. Our studies indicate that the additional refrigeration to increase the plant capacity by 50 percent could be installed in our plants for about half the cost that would have been required by a relative increase in oil rate.

In addition to the savings in first costs for a refrigeration system cooling the lean absorption oil, there is also a very definite saving in operating costs. The fuel requirements of a refrigeration system are practically limited to the engine fuel, while the fuel required to both heat and pump increased quantities of absorption oil would amount to considerably more. Also the other operating and maintenance costs are low in comparison with the costs for the additional boilers, stills, pumps, exchangers and their auxiliaries.

### Refrigeration Complements Absorption

Refrigeration units can be designed to maintain minimum oil temperatures where the plant is overloaded and can utilize the lowest temperatures that can be secured in cold weather. Where the temperatures that can be secured under this arrangement are lower than necessary or where freezing or hydrate formation occurs, a back-pressure valve, or temperature-operated motor valve can be installed in the vapor line from the chillers. In cases where constant plant operating conditions are desired, a temperature controller on the oil will be useful. This may be especially true where the existing absorption plant is overloaded at some one point such as the stabilizer. In such a case a controlled oil temperature may help maintain uniform vapor volumes and quality of products that would smooth out these plant operations.

In fact the refrigeration unit complements the absorption unit unusually well since it supplies the maximum cooling when the atmospheric cooling is the poorest. When the atmospheric cooling is less efficient and the temperature of the lean absorption oil to the refrigeration system is relatively high there is an increased temperature difference between the oil and the propane that results in increased cooling. This additional cooling in turn increases the volume of vapors to be handled by the compressor and therefore raises the propane pressure and temperature but not sufficiently to entirely offset the increased cooling. The fact that the compressors are more efficient with a higher intake pressure is advantageous to increased refrigeration. A compressor with clearance pockets has a still higher ratio of efficiency at higher intake pressure. The resulting lowered compression ratio allows its greatest advantage and maximum refrigeration at those times when the absorption plant, with its atmospheric cooling, is the least efficient.

The refrigeration unit supplements the absorption plant in still other ways that are important although not as generally appreciated. First, the fat oil entering the plant exchangers on its way to the preheater is proportionally lower in temperature as a result of refrigeration. This increases the mean temperature difference within the plant oil-to-oil exchangers and lowers the lean-oil temperatures to the plant coils. As a result these coils are required to extract less heat and a closer approach of the lean oil to the water temperature is realized. This reduced lean-oil temperature may be very appreciable in plants that are overloaded as is often the case when new refrigeration units are to be installed. On the other hand we have found relatively small increases in the heat load of the oil preheaters to the still.

Also the residual dry gas content, that is due to the vapor pressure of the light fractions in the lean oil, is reduced.[3] With the lowered oil temperature and corresponding drop in vapor pressure, there is a smaller loss of gasoline in the dry gas. This same factor would allow the use of a lighter mol weight absorption oil and resulting increased absorption efficiency since complete stripping in the plant still is not as essential.

Still another advantage of refrigeration results from the lowered dew point of the dry gas and the reduced water-vapor content. As a result there is usually no condensation in the dry-gas-fuel lines and this allows better performance through decreased pressure drops and reduced corrosion problems.

Another benefit lies in the fact that propane liquid can easily be made available for other services. These may vary from laboratory refrigeration, to air conditioning, to overhead condensation in a stabilizing column or to wet-gas cooling. Wet-gas cooling is not too important in a low pressure plant since higher exchange rates can be secured on the oil side and it is generally economic to take the full subcooling at this point. However for maximum refrigeration wet-gas cooling is definitely economic.

### Plant Design Simple

The process design of a refrigeration plant is fundamentally very simple. By the use of some practical data the operating conditions in a propane refrigeration plant can be found to a degree of

**TABLE 1**

**Propane Analysis—Mol Percent**

| | A | B | K | D | E | F |
|---|---|---|---|---|---|---|
| Carbon Dioxide.... | 2.10 | 0.30 | 7.00 | 0.00 | 0.15 | 0.10 |
| Methane..... | 7.35 | 0.51 | 14.41 | 0.00 | 0.19 | Trace |
| Ethane..... | 7.69 | 2.88 | 2.67 | 0.04 | 2.33 | 1.23 |
| Propane..... | 82.70 | 95.95 | 0.86 | 98.54 | 96.95 | 98.30 |
| Iso-Butane..... | 0.16 | 0.36 | 0.44 | 1.03 | 0.38 | 0.37 |
| Normal Butane.... | 0.00 | 0.00 | | 0.39 | 0.00 | Trace |
| Total..... | 100.00 | 100.00 | | 100.00 | 100.00 | 100.00 |
| Specific Gravity.... | 1.421 | 1.531 | | 1.551 | | |

**REMARKS**

A—Condenser Accumulator Vapor—172 #/sq. in. abs. & 79° F.
B—Condenser Accumulator Liquid—172 #/sq. in. abs. & 79° F. "Plant Propane" before any bleeding—vapors to compressor.
K—Equilibrium Constants, A/B.
D—Liquid in Chiller—lube oil removed.
E—Condenser Accumultor Liquid—162 #/sq. in. abs. & 79° F. "Plant Propane" after bleeding approximately 10 percent off B.
F—"Plant Propane" from Signal Hill Plant.
Specific Gravity taken by Edwards Balance.
A to D samples secured at Huntington Beach before bleeding and E after bleeding.

accuracy well within the range of the basic assumptions of future operating conditions in the plant. We also believe that any natural-gasoline plant can secure a satisfactory propane refrigerant from their plant.

When appreciable amounts of ethane and lighter contaminants are in the feed to the refrigeration system it is advisable to vent sufficient vapor from the condenser accumulator to bring the refrigerant down to a point of reasonable purity. This venting, or bleeding, is suggested in order to reduce the working pressures as well as the compressor horsepower. By limiting the probable discharge pressure the compressor cylinder can be rated for greater capacity. Since the vapor to be bled is at the bubble point, it can be shown that relatively small quantities of vapor need be removed, with the best results secured by bleeding this equilibrium vapor slowly (see actual analysis data in Table 1). For any temperature, vapor should be bled until the pressure is reduced to its allowable maximum (Figure 2).

We call the propane refrigerant that reaches this vapor pressure limitation a "plant propane." This is the type of material under consideration in this paper. Unless the propane feed is consistently very pure, the "plant propane" should represent an average propane refrigerant.

Another point that is important in considering the use of "plant propane" is the fact that the light ends in this refrigerant are flashed upon expansion into the chilling unit at a greater rate than the propane itself. The bulk of the propane requires additional heat before flashing into the vapor phase. As a result there is a greater proportional circulation of these light ends than there is of the pure propane. In actual operation the analysis that is secured at the intake to the compressors will show more of these light fractions than will be in the average propane in the chillers or in the system (Table 1). The extent of this condition is dependent upon the relative volume of propane in comparison with the vapor space in the same system and to the rate of circulation of the refrigerant. A large system and a light load could easily result in a circulation of much higher percentages of light ends than would be secured at high circulation rates. The analysis we have used for "plant propane" takes into consideration the effect of the average rate of circulation of these light ends and can be used for typical operations without unreasonable errors.

For these analyses, previous curves and data, a practical Mollier diagram has been developed (Figure 3) which has been adjusted for actual operating values. This enthalpy chart shows the amount of heat in a pound of the "plant propane" plotted against pressures, with lines of constant temperatures shown between the lines of saturated liquid and saturated vapor. These have been corrected for the light ends in the "plant propane" mixture so that they follow a curve of boiling points between the saturated liquid and vapor limits. From this diagram it is possible to determine the relative amount of heat and the pressures and temperatures that can be expected at any point in the operation. The diagram will show the amount of heat to be removed in the condensers, the effective cooling available in the chillers, the theoretical horsepower of compression and many more important values. In fact, it will be the engineers' "bible."

**CONDENSER, PRESSURE-TEMPERATURE CURVES**

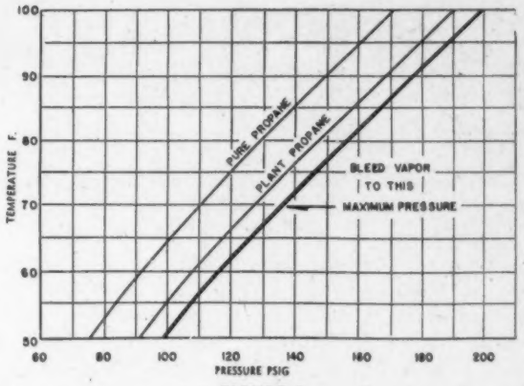



**FIGURE 2**

**Curves indicate allowable pressures for a given temperature secured by bleeding light fractions from propane.**

**MOLLIER DIAGRAM-"PLANT PROPANE"**

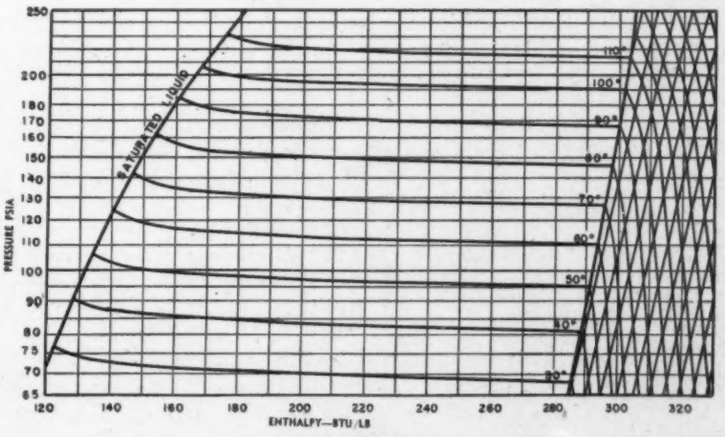



**FIGURE 3**

**Mollier diagram for "plant propane" based on pure propane and adjusted for operating data.**

We are including a typical flow diagram for a refrigeration cycle on lean-oil subcooling (Figure 4). This is offered as a guide to the general nomenclature

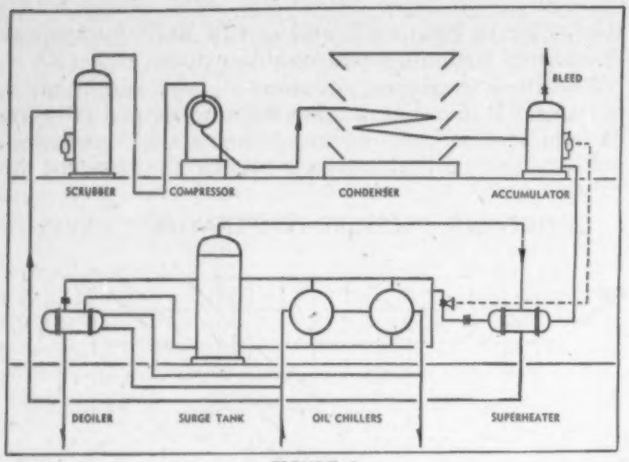



**FIGURE 4**
**Typical Flow Sheet**

and as a base for more detailed discussion on the design of various units. This flow diagram is not submitted with any recommendation that this arrangement is preferable to others. It is only one of several. For example, our condensers are fabricated pipe coils but actually open sections or a closed condenser would be just as suitable. Also, the run-down accumulator can be designed for greater capacity and allowed to take any fluctuations in the liquid propane in the system. In such a condition the level control would be on the chiller unit instead of the accumulator. It is important to study the condenser under conditions of poorest cooling to determine the value of added surface against the horsepower of compression. The Mollier diagram will show the temperature-pressure relationship in the condensers and the special curve (Figure 2) should show these values even more clearly. This change in pressure with the condenser temperature must be used in determining the relative values of added surface and additional compressor capacity.

Oil chillers generally are constructed in one of two types. First, a chiller without any vapor space in it which operates in conjunction with a surge tank. Second, a type with a vapor space that allows a level to be maintained within the unit. In either case the size of the chiller should be balanced against the compressor capacity, since within reasonable economic limits the same cooling can be secured by either means.

The lubricating oil from the compressor cylinders must be removed from the system before its volume builds up to the point that temperatures are affected or the liquid begins foaming. It is not felt to be economic to take any steps to remove the oil from the vapor at the compressor discharge since sufficient oil will probably get by and into the liquid propane to require its removal at this point. Actually a pure mineral lubricating oil offers no operating problem and a deoiler can be installed to accomplish its removal before any excessive concentration is possible. A side stream of the oil or other material to be cooled may be used for heating and since the cooling effect of the propane may be utilized there is no economic reason for not designing the deoiler rather liberally. It must be remembered too that the lube-oil level must be kept low since the foaming oil will otherwise carry over with the vapors.

### Design of Superheater

It is felt that the utility and the design of the superheater deserves more consideration than is generally accorded it. This unit not only superheats the vapor and prevents condensation during compression but at the same time the subcooling increases the refrigeration in terms of horsepower per unit of cooling. With an impure, or "plant propane," there is an additional advantage that can be secured from this subcooling. A study of the enthalpy chart will show that subcooling the liquid will reduce the quantity of flash upon expansion and at the same time will also reduce the flash temperature of the "plant propane." If the chillers are designed to take advantage of this reduced temperature, and the increased temperature difference that can result, greater refrigeration can be secured without any change in operating pressures. This will increase the net refrigeration and offers some very interesting economic possibilities, especially if the available horsepower assigned to this service is limited.

We believe that the complete unit should be designed for 250 psi or the equivalent working pressure required for "plant propane" storage. Safety valves will be set to work at this pressure but except for fire they should never operate. With this allowable working pressure even the engines can be shut down without the necessity of releasing any of the propane from the system. The knowledge that the failure of any of the controls, or of the compressors, cannot cause a hazardous condition to materialize, allows a very healthy feeling of security on the part of all concerned. The cost of this added safety is nominal.

### Plants Better Performance

Propane refrigeration units have been added to two of Signal Oil & Gas Company's low-pressure absorption plants during the last year. At Signal Hill this allowed one plant to efficiently handle the wet gas previously treated in two plants. At Huntington Beach this addition was required to allow the plant to maintain its efficiency with the increased volumes of wet gas to be processed. The two units are naturally somewhat similar in their flow (Figure 4) but differ considerable in their construction, especially in the case of the oil chillers (Figures 5 and 6). Both plants have chillers on the total plant production as it is being pumped into the raw feed tank for the stabilizer. In addition, the Huntington Beach plant has a chiller on the natural gasoline and uncondensed vapors from the still run down condensers (Figure 9).

Both plants better their estimated performance and have provided no unforeseen operating problem or hazard. In fact, the only unusual conditions that we have had in a year or operation was the result of the failure of the air supply to the level control on the accumulator at Huntington Beach. This allowed the motor valve controlling the expansion of the liquid into the chillers to stand wide open and allowed all the liquid and a relatively small quantity of the vapors to pass into the chiler. The result was a small increase in the vapor load to the compressors and a decrease in the compressor discharge pressure of a few pounds, since full condensation was not required. The higher propane pressure in the chillers raised the oil temperature a few degrees but not enough to upset the plant operation and no hazard resulted from this failure.

A set of operating and performance data for both plants is included (Table 2). In general these are in good agreement with the Mollier diagram. For

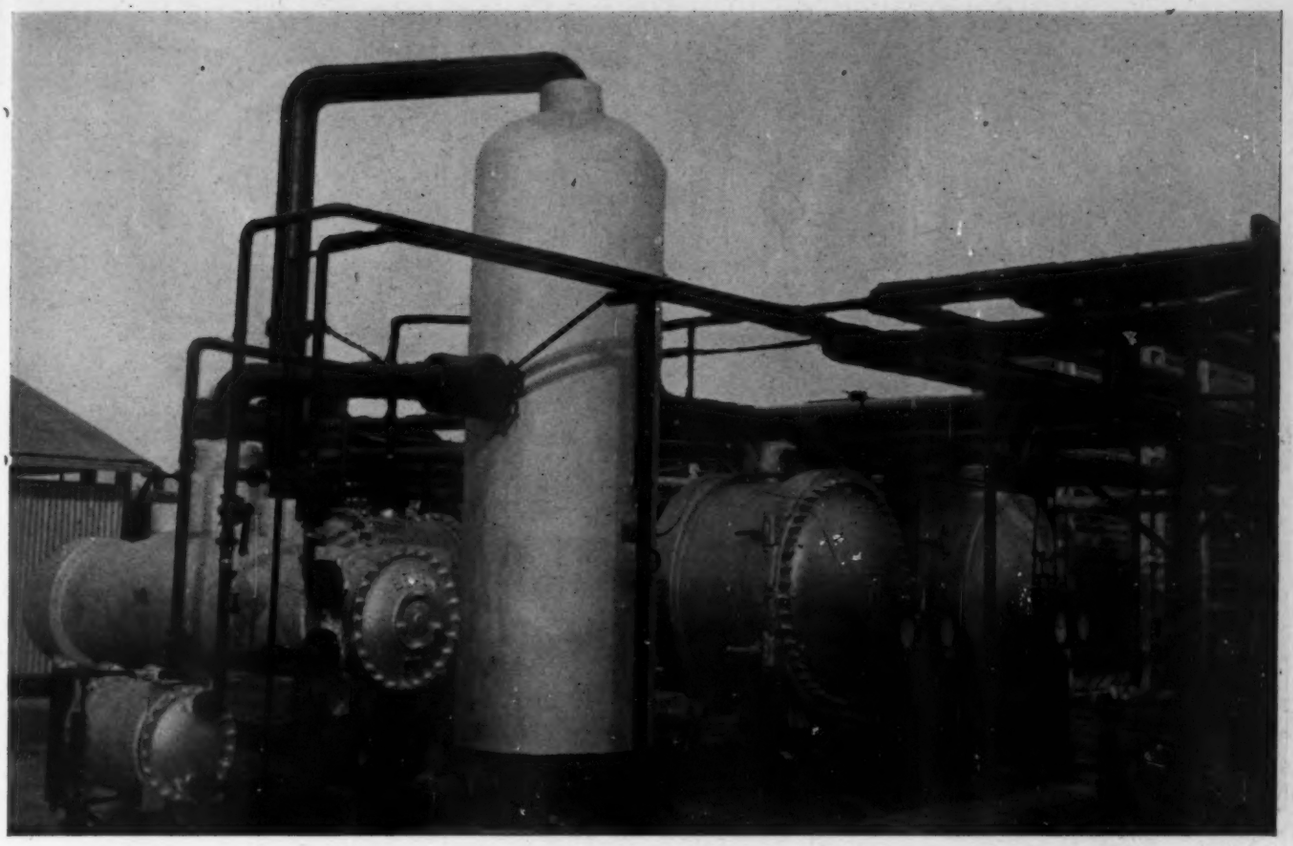


FIGURE 5

Signal Hill Refrigeration Unit. From right to left the units are deoiler, raw gasoline chiller, propane surge tank and oil chillers.

FIGURE 6

Huntington Beach Oil Chillers. The 39-inch by 65-foot unit at the left is the final oil chiller in series with the two 25-inch by 63-foot chillers on the right. The deoiler, superheater and the scrubber are shown between the two chiller units.

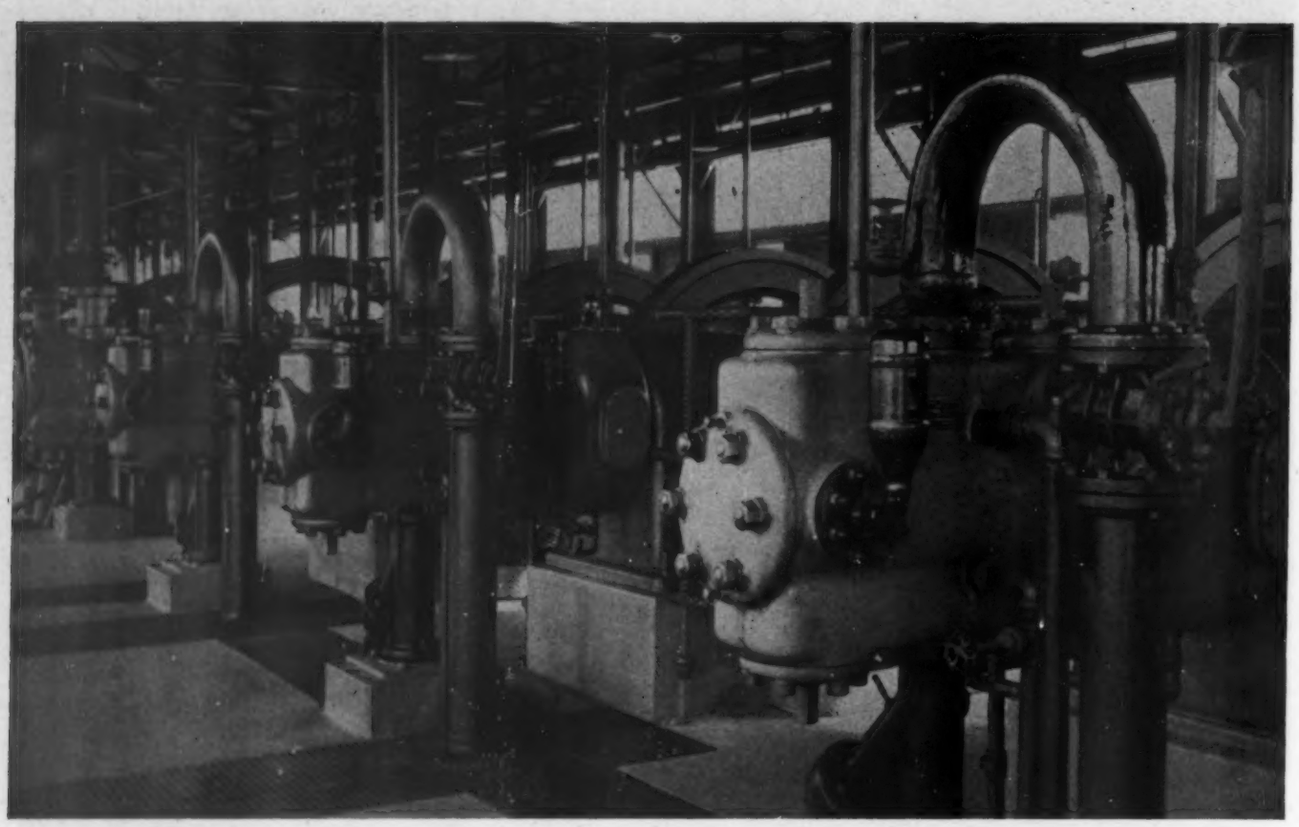
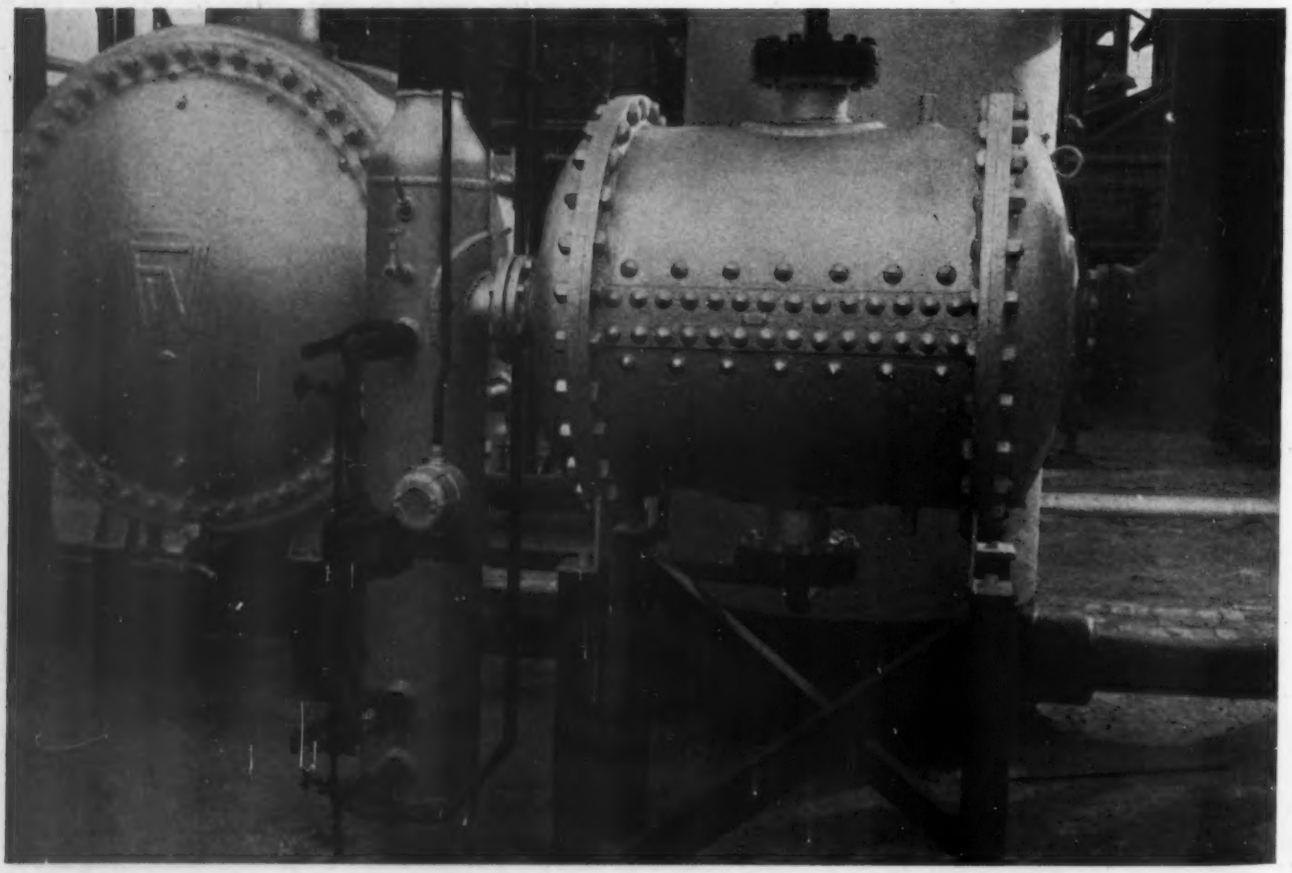


FIGURE 7
Huntington Beach Propane Compressors.

FIGURE 8
Signal Hill Superheater and Scrubber. The surge tank actually serves as a scrubber in this plant so that this unit serves as means of installing a float and magetno ground.



further comparison a set of the enthalpy data for Huntington Beach is given below:

| | Btu per Pound |
|---|---|
| Compression: | |
| Inlet vapor—94 lbs. abs., 61° | 296 |
| Discharge—174 pounds abs., 125° | 309 |
| Work of compression | —15 |
| Condensation: | |
| Inlet—173 lbs. abs., 117° | 313 |
| Outlet—171 pounds abs., 85° | 157 |
| Heat of condensation | 156 |
| Superheater: | |
| Liquid in—171 pounds abs., 85° | 157 |
| Liquid out—Saturated liquid, 80° | 153 |
| Subcooling | 4 |
| Refrigeration: | |
| Liquid in—98 pounds abs., 48° | 153 |
| Vapor out—98 pounds abs., 53° | 292 |
| Net refrigeration | —139 |

The data taken from the Mollier diagram agrees with the actual test data very closely except for the compressor performance. In this case the Mollier diagram gives a 108° F. discharge temperature while it was actually 125° F. Also, the 15 Btu per pound for the theoretical work of compression is too low a value. Both differences are due primarily to the fact that our intake and discharge pressures are secured from pipe-line connections. Actually the pressure losses through the compressor valves will result in a lower intake and a higher discharge pressure within the cylinder and therefore a greater ratio of compression and increased theoretical horsepower. Since the ratios of compression for this work are generally about 2.0, or less, a small change in either pressure can result in a relatively large percentage increase in horsepower. Also, the jacket cooling water is generally warmer than the propane to the cylinder and therefore gives up heat in the cylinder and heats rather than cools the vapor.

This difference in readings is typical and must be considered in any design before any value is accepted for the compressor horsepower. The actual horsepower required to compress propane is less than generally required for natural gases due to the relatively low ratio of specific heats (1.17). For this reason one of the more generally accepted curves of horsepower per million cubic feet per day for different ratios of compression, can be used[4] or any other curve or empirical data with which you are familiar and can interpret for this type of service.

Continuing with our discussion of our actual operating and design data, there are several other points of interest in connection with the compressors. These include the use of a non-return or a check valve which should be installed on the discharge of the compressor. We also feel it it better to install the compressor-intake line so that it rises and makes a 180° bend with the shut-off valve on the riser. This reduces the possibility of liquid accumulating above the intake valve during periods when the compressor is down (Figure 7). In cases where the shut-off valve must be placed in a position that would allow the accumulation of liquid between the valve and the header it is very important to install a bleeder that will permit complete drainage of any liquid before the engine is started.

We believe that there are a number of advantages favoring the use of a slightly oversize compressor

**TABLE 2**
**Operating Data**

| | Signal Hill | Huntington Beach |
|---|---|---|
| General: | | |
| Oil Rate—1000 gals day | 585 | 1,110 |
| Natural gasoline—gals day | 28,500 | 45,500 |
| Propane Refrigerant: | | |
| Mcf day | 3,260 | 5,520 |
| Lbs hr. | 15,800 | 27,000 |
| Gal min. | 62 | 105 |
| Condensers: | | |
| Surface (O. D.)—sq ft. | 3,670 | 5,810 |
| Pressure —in. | 152 | 173 |
| —out. | 148 | 171 |
| Temperature —in. | 106 | 117 |
| —out. | 76 | 85 |
| Superheater: | | |
| Surface (O. D.)—sq. ft. | 385 | 250 |
| Temperatures: | | |
| Vapor—in. | 46 | 53 |
| —out. | 58 | 60 |
| Liquid—in. | 76 | 85 |
| —out. | 67 | 80 |
| Oil Chillers: | | |
| Surface (O. D.)—sq. ft. | 2,660 | 7,650 |
| Propane—gal. min.: | | 93 |
| Pressures: | | |
| Oil—in. | 92 | 72 |
| —out. | 84 | 65 |
| Chiller | 88 | 98 |
| Temperatures: | | |
| Oil—in. | 74 | 73 |
| —out. | 49 | 53 |
| Propane—Flash | ..... | 48 |
| —Final | ..... | 53 |
| —Surge | 46 | ..... |
| Gasoline Chillers: | | |
| Surface—sq. ft. | 450 | 450 |
| Pressures: | | |
| Gasoline—in. | 105 | 105 |
| Propane | 88 | 105 |
| Temperatures: | | |
| Gasoline—in. | 87 | 82 |
| —out. | 63 | 60 |
| Propane | 46 | 55 |
| Compressors: | | |
| Pressures: | | |
| Intake | 87 | 94 |
| Discharge | 152 | 174 |
| Compression Ratio | 1.75 | 1.85 |
| Temperatures: | | |
| Intake | 58 | 61 |
| Discharge | 113 | 125 |
| Engine cooling water | 110 | 110 |

Remarks: One-third additional compressor capacity is being installed at Huntington Beach.
Pressures given in pounds per square inch, absolute, and temperature in degrees Fahrenheit.

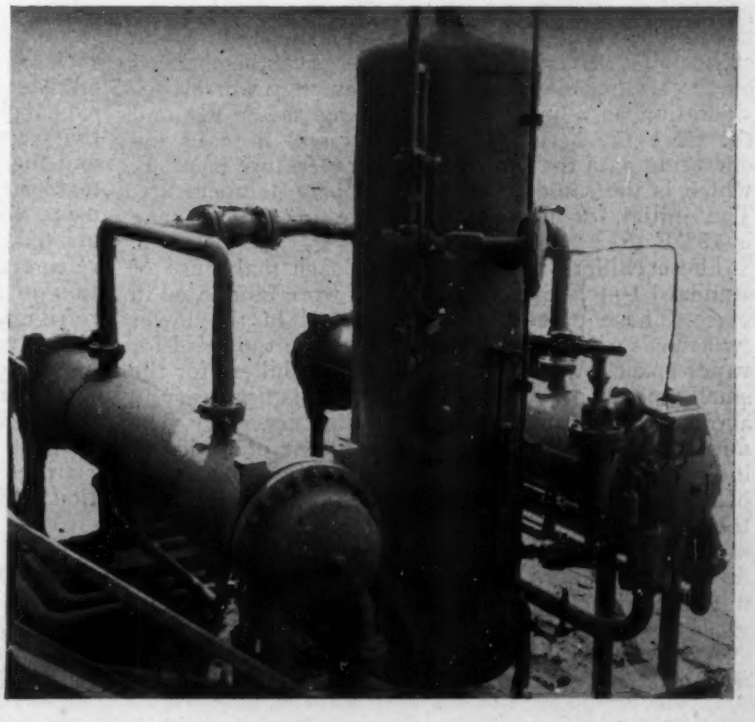


**FIGURE 9**
**Huntington Beach Gasoline Chillers. Note separate surge tank and level control for independent operation.**

cylinder and then bringing it down to the desired operating range by the use of clearance bottles. The most important of these advantages is the flexibility that is offered. This not only allows the correct capacity for the calculated design conditions to be secured but will allow a clearance adjustment to be made at any time to meet further or changed operations over a wide range of conditions. Also, clearance pockets have less effect on the cylinder capacity at low compression ratios such as exist under the higher intake pressures and higher loads. For this reason when the atmospheric cooling is the poorest and the chillers are producing the greatest cooling effect, the compressor cylinders are handling this increased volume of propane with the highest relative efficiency. This is an ideal situation since it allows the greatest load to result at those times when the value is greatest and higher horsepower can be justified.

### Condensers

The condensers in our plants are pipe coils designed for the necessary drainage to handle full condensation without unnecessary pressure drop. It can be seen from the Mollier diagram that there will be a relatively large mean temperature difference (MTD) between the cooling water and the propane except for the last ten percent, or less when the "plant propane" temperature curve swings up as a result of the light hydrocarbons which reduces the temperature difference very materially. Actual test data indicates that half the heat of condensation was done in the first 30 percent and two thirds in 50 percent of the coil.

### Oil Chiller

In the Huntington Beach plant the oil chiller was designed to utilize the range in boiling temperatures available in a "plant propane" at a constant pressure. The oil chillers at this plant consist of one 39-inch by 65-foot and two parallel 25-inch by 65-foot long units in series flow. The propane is expanded into one end of the 39-inch chiller and travels the length of this unit before it overflows a weir that maintains the level of the propane and flows into the other chiller. The chillers are piped for counter flow. The propane temperature increases about 5 degrees between the point of expansion and the end of the chillers. These changing temperatures can be checked on the Mollier diagram and are obivously worth consideration as a means of increasing the MTD and therefore the actual cooling. You will note in the operating data that our final oil temperature is 53° F. which is the same as the final propane temperature. The initial, or flash, temperature of the propane is 48° F.

These chillers at Huntington Beach make use of standard 1-inch pipe for tubes and were fabricated in place. These pipe tubes were placed in the lower two thirds of the shell which leaves the top third for vapor space. We also utilize these oil chillers for the small amount of working storage that is necessary to avoid adding propane more often than necessary. The minimum level of propane for efficient operation is indicated by a superheating of the vapors as a result of the direct contact with the tubes as the level drops too low. This minimum efficient level can be determined definitely if thermometers are installed in both liquid and vapors. This will then give a reliable minimum level that can be indicated by a gauge-glass reading during normal operation.

At Signal Hill, commercial exchange units were installed. They have performed as well as could be desired. However, with this type of design, which is very similar to that shown in the flow diagram, there is no means of taking efficient advantage of the lower flash temperature and slightly greater surface is required for the same refrigeration. The economic choice between these two types of cooler will depend upon individual conditions and this can be decided after considering both possibilities.

### Deoiler

The deoiler must secure its feed from the point where there will normally be the greatest concentration of lubricating oil in the system. At Signal Hill where the propane is in constant circulation the supply is taken from the surge tank. At Huntington Beach the propane enters at the end of a long horizontal chilling unit and flows lengthwise through this unit. For this reason the lubricating oil will become concentrated at the end of the unit and the feed for the deoiler is taken from this point of greatest concentration. In this plant where the design allows the lubricating oil to concentrate within the system, the problem of removing the oil is obviously simplified.

To further describe the units, at Huntington Beach we have chillers on both our run-down gasoline and uncondensed vapors and on the raw production into the stabilizer feed tank (Figure 9). These units are independent of the oil chillers and are located in another area of the plant. Both are converted exchanger units and are connected top and bottom to a propane surge tank. A level-control on the surge tank maintains an independent level in this tank. A small drain line to the deoiler is used to remove the lubricating oil that concentrates in this unit.

The chiller unit on the run-down liquid and uncondensed vapors from the still condenser is used to control the volume of vapor which must be handled by the vapor compressor. The other chiller unit is on the plant production to the stabilizer-feed tank and in this case the cooling permits a lower presure for separation of the excess light fractions. This lower temperature allows a better separation of the light hydrocarbons because of their relatively smaller reduction in vapor pressure at these colder temperatures.[5] The amount of cooling on each chiller is regulated manually by an occasional adjustment of the valve on the vapor line out of the exchanger and into the surge tank. It has been found that very even control can be secured by this means and the back-pressure valves or controllers were not necessary. The addition of these two chillers allowed us to continue with the same vapor compressor as existed in the plant before the addition of the refrigeration unit and the resulting increased extraction of light hydrocarbons. It was found that the refrigeration horsepower necessary to reduce the vapor volume and lower the discharge pressure is approximately 60 percent of the horsepower that would have been required to handle the additional vapors without this refrigeration. The additional 40 percent of the horsepower is available for additional refrigeration. There has been no problem in connection with this operation.

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## Selection, Installation and Maintenance of

# Refinery Process Control Instruments

### Part 1

### Flow Metering and Liquid Level

EUGENE BAINBRIDGE

INSTRUMENTS commonly used in an oil refinery can be classified into four general groups: flow meters, liquid-level recorders, pressure gauges and temperature instruments. Any one of these cannot be selected as being more important than the other, and if more information is given in this series about one than another, it is because more information is available or the instrument itself requires a much clearer discussion.

The flow meter is a common tool in the refinery and it was only after the close measurement and control of flow was introduced that the present highly complex processes were possible.

In selection, installation and maintenance of equipment the first task is to select the proper type for the job.

Generally speaking, there are three choices, the differential meter, the area-type meter and the positive-displacement meter. The selection of the meter for the job is fairly easy because of the characteristics of each type. For most refinery applications the differential meter using the thin-plate orifice will be selected. There are many good reasons for this choice. It is easy to use as a recorder, and it is very easy to adapt to control processes; it is easily changed from a low volume to large volume, without an expensive maintenance problem; the size of the pipe does not determine the cost of the equipment, and the meter can be used on installations other than the original specified job.

But, this type has several disadvantages, and not the least of these is the necessity of protecting the internal mechanism from any corrosive liquids or gasses which might be present in the flowing medium. This problem with its accompanying seal pots and necessary piping, gives the instrument-maintenance department many headaches. The instrument is less accurate at low flows, because of the friction in the bearings and the power to overcome this friction comes from the rise and fall of the mercury in the float chamber. This rise and fall becomes progressively smaller as the flow becomes less, because the rate of flow varies as the square of the differential pressure. Therefore, there is less power at the lower flows and the friction ratio increases, decreasing the accuracy of the meter.

The choice of one of the other types is generally made because of smaller initial cost, and it can be used where a recording and controlling mechanism is not desired.

The rotameter type has certain advantages. It is easily made corrosion proof; its visible float is a check on the correct operation of the meter; it has uniform flow graduations; a fixed pressure loss over the entire range of the meter, and the capacity of the meter can be changed by changing the weight of the float.

Here again in this type we find disadvantages. The glass tube is fragile and if subject to physical or thermal shock would easily break; opaque fluids are not easily measured, and the instrument requires frequent cleaning.

Another area meter is the piston type. It generally is used in applications where the fluid has a high viscosity. An advantage is that it incorporates an induction-bridge system of transmitting the flow indication to a remote receiving instrument with possibility of additional ease of recording and controlling.

The third type, the volumetric meter, generally is used in the measurement of gasoline and other light oils at loading stations. It is also becoming more common on transport trucks where it is desired to measure gasoline to service outlets. This type of meter has the advantage of being acurate on small rates of flow. It is not affected by changes in pressure, density or viscosity; the installation costs are low; they are compact and portable in the smaller sizes, and they do not require level conditions to maintain accuracy. Disadvantages are that they are very expensive in larger sizes; dirty or abrasive fluids cannot be measured, repairs are relatively expensive; they lack flexibility—cannot be changed from small to large pipe, and calibration is difficult and tedious.

It is evident that the selection of the meter depends entirely on the application and that each type has advantages that make it preferable to the others when placed in the proper application.

#### Installation of Flow Meters

Installation of flow-measuring meters is relatively simple in the volumetric and rotameter types. In the volumetric type, all that is necessary for proper installation is to provide some manner of protection against abrasive material entering the meter and to see that the meter is installed with the inlet and outlet in the right location. The installation of the rotameter must be made with careful consideration given

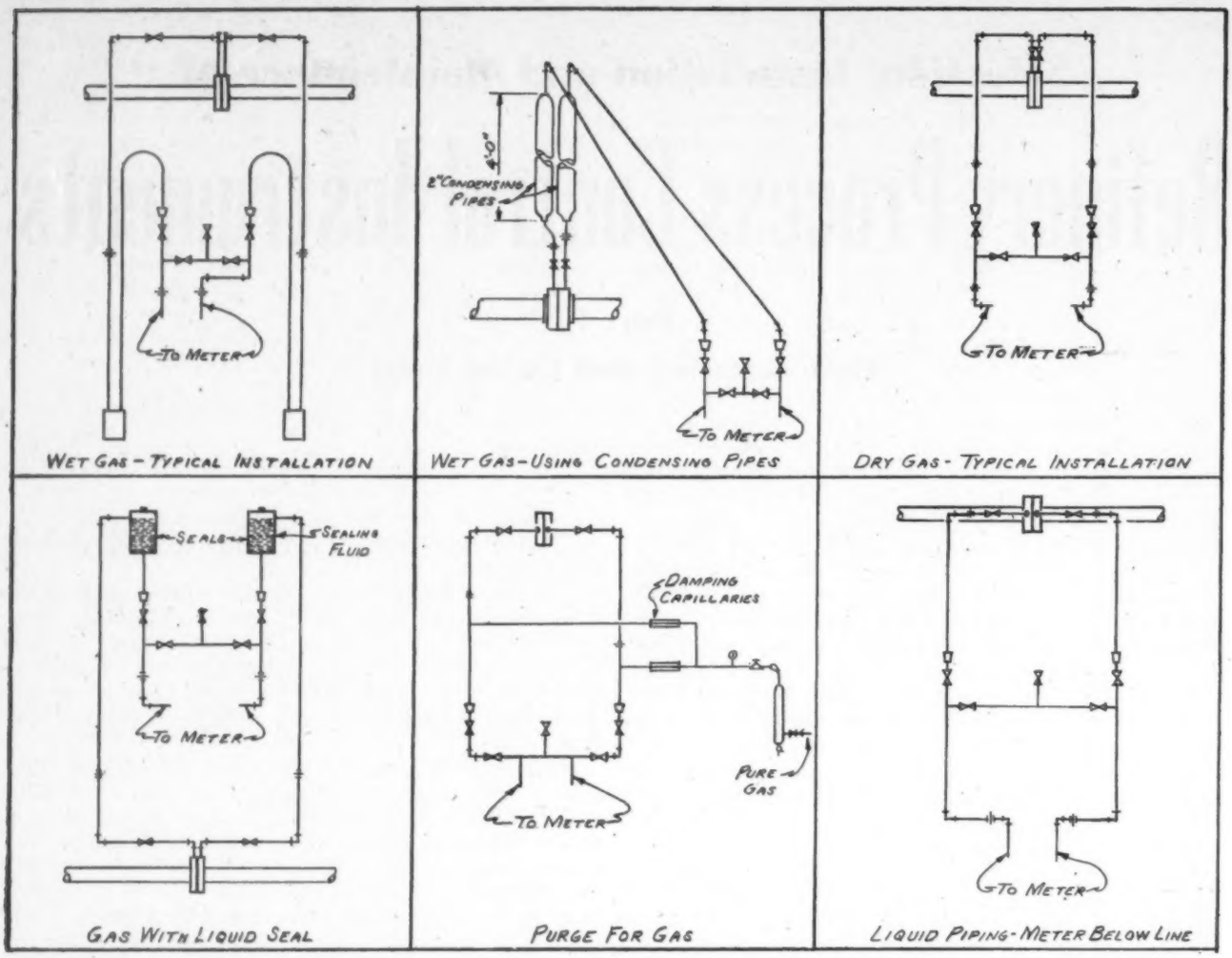



FIGURE 1

the bracing of the meter, as, if this is not properly done the glass will be subject to shock and consequent breakage. Often a stress due to piping distortion will result in trouble, with either breaking of the tube or packing leaks. Attention need not be paid to the number of straight runs of pipe before or after this type, but care should be used if the piston type is installed as this meter requires 20 pipe diameters of straight pipe before the meter.

Utmost care must be used in the installation of the differential type meter to insure maximum accuracy. This is especially true where the characteristic of the fluid being metered necessitates use of seal pots or a purge system.

The principle of the differential meter involves the use of a primary element to create a differential in pressure at two different points in the line. This primary device can be one of several general groups—it can be the thin-plate orifice, the venturi tube, the flow nozzle, or the pitot tube. Each of these has advantages and disadvantages. The most commonly used is the thin-plate orifice, and a study of the characteristics of each of these will show the reason.

The venturi tube has the highest accuracy and pressure recovery is greater than in any other type, and its construction obviously will resist abrasion and accumulation of dirt or sediment on its upstream opening. It has the disadvantages of being costly, and can not be duplicated in the shop, neither can it be changed easily and is not adaptable to other than the conditions for which it was purchased.

The flow nozzle has the general advantages of the venturi tube to a lesser degree and is cheaper. It also is not adaptable to other than its original installation.

Either the venturi or flow nozzle has fine accuracy with high ratios to pipe size. They will operate with d/D ratios greater than 0.75.

The pitot tube has a definite place in the operations of a refinery because it can be used to measure flow where a temporary installation is necessary and also where the size of the pipe is too great to make an orifice installation practical. The objection to the pitot tube is that it cannot be used for any length of time in a fluid containing dirt or solids of any kind. Any foreign materials will either clog the tube or build up on the exposed surface to such an extent as to seriously change the calibration of the instrument.

The big advantage of the thin-plate orifice over other types of primary elements is its relatively easy duplication and consequent low cost, but there are other reasons for its popularity. It is easily interchanged and a new flow condition can easily be corrected for; complete data are available for computing flow; little room is required, as it can be inserted in available flanges with little changes. The disadvantages include low efficiency, the pressure recovery is lower than for the other types; poor accuracy on high d/D ratios; subject to wear, causing

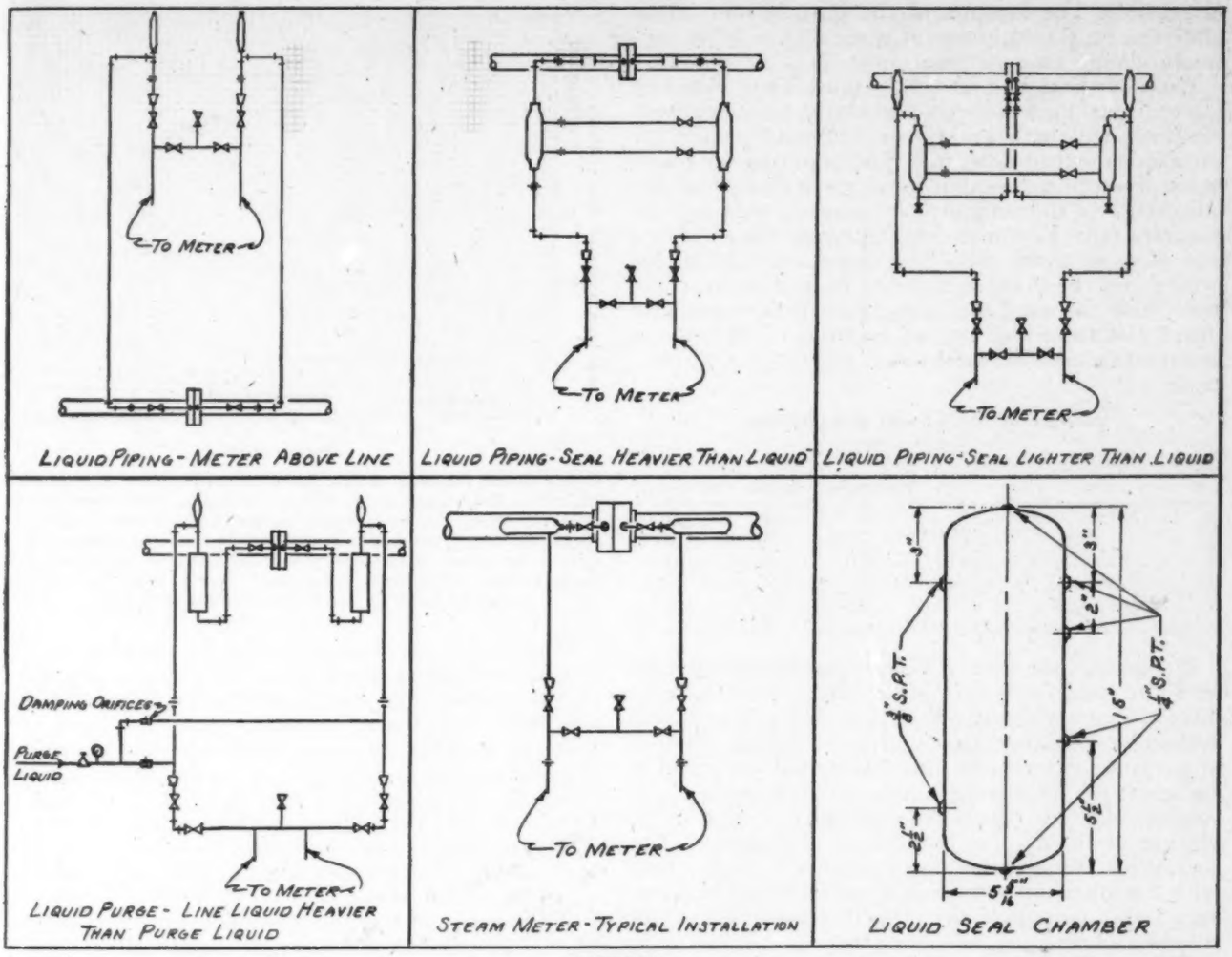



**FIGURE 2**

inaccuracies, obstructs flow of dirt or sediment in pipe which can affect the accuracy.

The orifice plate can be placed in either horizontal or vertical runs of pipe if certain precautions are considered. Installation of the piping to the meter can be most easily presented by showing a series of typical installations under various conditions (Figures 1 and 2). These are self explanatory and will take care of installations in the average refinery.

There are certain precautions necessary when piping a meter. The diameter of the pipe from the meter body to the orifice should be ½-inch for a distance less than 25 feet, ¾-inch for distances between 25 and 50 feet, and over 50 feet 1-inch pipe should be used. Some instrument departments use ⅜- and ½-inch copper tubing for piping steam meters. Long distances are not recommended because of the lag due to friction, and if it is necessary to locate the meter some distance from the orifice, a system of telemetering should be used. Lead lines should be self venting and self draining, therefore, a slope of at least 1-inch to the foot to the meter should be used.

The meter should be installed level, as this is a common source of error. All high spots in the piping should have vapor pots, especially if the fluid is subject to vaporization. Likewise, if there is any possibility of sediment being present, all low spots in the piping should be equipped with sediment pots.

As a general rule gate valves should be used at orifice taps. For the instrument manifold at the meter, needle valves can be used. A five-valve manifold makes possible a positive check on the equalizing valve.

The seal piping system should be equipped with large seal pots to reduce the displacement of sealing medium to a minimum. It should be remembered that when the instrument is in service, opening of the equalizing valve without closing an orifice block valve will result in a blowing of the seals. The seal liquid should be heavier than the flowing liquid. Where acids are encountered it usually is necessary to use a seal liquid lighter than the flowing liquid. This involves the use of a seal piping assembly somewhat different from that used in oil applications. The sealing liquid commonly used on acids is kerosine. Sometimes the usual piping arrangement can be used for weak acids by using special oil. As a general rule steam and water installations are steam traced to keep them from freezing. Lately, dibutyl-phthalate has been found to be more satisfactory for use as a sealing medium for these installations.

In the case of the liquid purge system, gas or air can be used to purge the meter piping and in the case of gasses an inert gas should be used. The piping of the purge system includes orifice unions to properly distribute the purging medium.

When using the venturi tube, pitot tube or flow nozzle, the pressure tap location has been chosen by the manufacturer, while in the orifice plate a choice

is possible. The location of the taps for the orifice plate can be placed in one of three ways—Vene contracta, flange taps, or pipe taps.

Each of these has its advantages. The vena contracta, if installed properly will be the most accurate. As there will be a greater pressure differential for each increment of flow, this type produces the maximum pressure differential for a given flow. The disadvantage is the difficulty in properly locating the pressure taps. The upstream tap centerline is located one pipe diameter from the upstream face of the orifice and the downstream tap centerline at a distance from the same upstream face which varies with the d/D ratio. The proper location of the downstream tap can be determined from the following table.

**Location of Downstream Pressure Tap**
(In Pipe-Diameters)

| d/D | Minimum | Mean | Maximum |
|---|---|---|---|
| .20 | .37 | .86 | 1.30 |
| .30 | .44 | .80 | 1.15 |
| .40 | .47 | .73 | 1.00 |
| .50 | .47 | .66 | .84 |
| .60 | .42 | .57 | .70 |
| .70 | .34 | .45 | .55 |
| .80 | .22 | .33 | .41 |

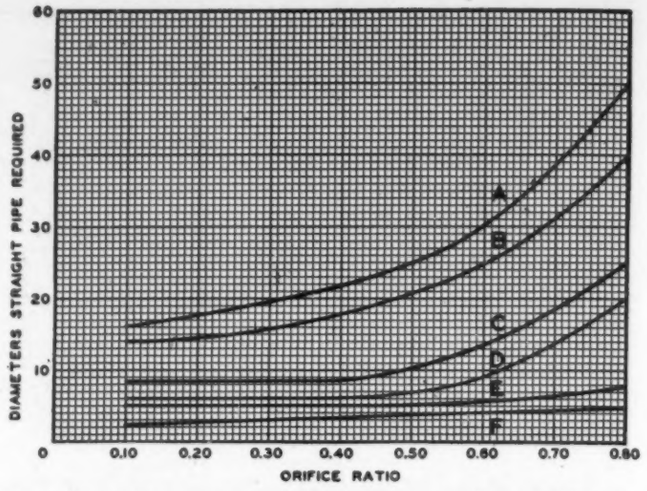
Flange taps are located 1 inch from the orifice plate or 15/16-inch from the gasket face. This distance does not vary with the size of pipe or the orifice ratio. Although the flange taps sacrifice a certain amount of pressure differential, the differential produced is sufficient for measuring the flow. An important advantage of flange taps is the possibility of error in the placing of the taps is reduced to a minimum. The cost of installing flange taps is generally higher than with the other types because a special flange is necessary and in large pipe sizes this increased cost can be considerable.

The third type of tap installation, pipe taps, is used on small piping where the size prohibits accurate placing of flange taps and also on larger piping where initial installation cost is a factor. Pipe taps can be used where a large flow must be measured with a meter of a limited range. In this case the lower differential will permit a smaller ratio of orifice-to-pipe diameters. Pipe taps are located 2½ pipe diameters upstream and 8 pipe diameters downstream. A small error in the placing of the taps is permissible.

Certain minimum requirements for straight piping, both preceding and following the orifice must be observed to avoid the effects of swirls, cross currents and eddies. In some installations, where absolutely necessary, straightening vanes are recommended, but because of the possibilities of these becoming detached from the line and causing an error they should be avoided where possible. The accompanying curve shows minimum requirements of straight piping for a proper installation.

Since, in larger plants the orifice plates are made in the machine shop, we should consider its construction. The plate thickness is guided by the strength required to prevent buckling under ordinary differential pressures. This is usually ⅛-inch in the smaller sizes and ¼-inch in the larger pipe. The edge thickness must be within the following limits:

1. Not greater than 1/30th of the internal diameter of the pipe.
2. Not greater than ⅛th of the orifice diameter.
3. Not greater than ¼th of the orifice rim, D-d/2.




Curves showing the run of straight pipe necessary preceding and following the orifice. This depends on the orifice ratio and the number and type of valves or elbows. For the upstream run use: Curve A after a globe or regulating valve; Curve B after two elbows not in the same plane; Curve C after two elbows in the same plane; Curve D after one elbow; and Curve E after straightening vanes. Use Curve F after the metering element itself, i.e., for the downstream run of pipe.

If the thickness is greater than the specified limits, the plate must be bevelled on the downstream side, and the sharp edge side should be marked "UP" on the orifice tab, which should be on all orifices. The material used for the orifice should be as resistant as possible to corrosion, as one of the important factors affecting corrosion is the velocity of the fluid, and it is evident that the edge of the orifice will be subject to the maximum corrosion rate, therefore, as a general rule the orifice plate should be made of a material superior to that of the pipe.

The additional care taken in the installation of flow meters in general will be well worth while, as the percentage of error due to improper installation is unpredictable and not constant and therefore no way of determining or correcting for this error is possible.

Among the common errors found in the installation of the primary elements and piping are the following:

1. Improper centering of the orifice plate.
2. Orifice with the bevelled edge upstream of the flow.
3. Gaskets at orifice projecting into the pipe.
4. Faulty installation of taps, either nipple projecting into the line or burr left by drilling and tapping operation.
5. Leaking pressure line, causing false differential.

### Maintenance of Meters Simple

The problem of maintenance in the average refinery as far as flow meters are concerned is simple. Dirt is the common enemy of all types and it is obvious that a dirty meter, whether it be any of the types previously discussed, will have to be cleaned. The important thing is to determine when the meter should be taken out of service and overhauled.

In the rotameter, necessity for cleaning is obvious, due to the glass tube which incloses the float getting dirty. And similarly, a dirty condition is indicated in the displacement meter, when the fluid being measured does not pass through the meter and a partial or total restriction of flow results. The remedy is to take the meter apart and clean it. Very seldom will it be necessary to replace parts in either of these meters. In the displacement meter, if the flowing fluid should

carry any quantity of abrasive materials, the discs or buckets that have recommended clearances should be carefully inspected, as any excessive leakage will result in a serious error, and at smaller flows this leakage could become prohibitive. Since calibration of the rotameter depends to a great extent on the weight of the float, this part should be checked often any sediment collected on the surface should be removed.

Maintenance problems of the orifice or differential meter is a little more difficult. Here we do not have the advantage of such obvious meter trouble as in the two preceding types. As a general rule it is not recommended that meters be given a periodical overhauling, unless the meter is installed in an application where previous experience has shown the necessity. With the proper use of seals the meter body should remain in operation for long periods.

The necessity of taking the meter out of service for overhauling generally will be indicated by a sluggish action. It is good practice to have a regular schedule for testing meters with a water column, and proper records of the results should be kept. This will aid in determining when a meter should be taken out of service. A definite schedule of checking the meter to zero should be used, as this will normally show up an error due to improper seals, loss of mercury or a binding pressure-tight-bearing assembly. Although in most differential meters the amount of mercury is not critical, sufficient mercury should be used to give full-scale float travel and resulting pen movement.

The pressure-tight bearing or shaft is a regular source of trouble. Generally speaking this is about the most troublesome thing a meterman has to contend with in the maintenance of the flow meter. As the power to move the shaft is small due to the limitations of the float area, friction must be reduced to a minimum. Too much grease should not be used as this will result in a binding of the shaft, but a sufficient amount should be used to keep the fluid from leaking into the meter case. The electrical systems for transmitting the movement of the float to the pen mechanism has a definite advantage here, and where the possibility of escaping liquid or gas being injurious to the health of the operator or where the escaping gas is combustible, this type should be used.

It is essential that the equalizing valve does not leak. To check this, close the orifice block valves when meter is registering flow. If the pen moves, the equalizing valve is leaking. The five-valve manifold will reduce the possibility of error from this source to a negligible amount.

## Liquid-Level Instruments

SELECTION of liquid-level-indicating equipment depends on the characteristics of the tower or vessel in which the level is to be measured, as well as the characteristics of the liquid within. If the tower has obstructions on the outside, an internal-type instrument is required, and if the obstructions are on the inside of the tower then the external-type should be used.

There are many different types of liquid-level instruments and it is not within the scope of this paper to discuss all of them. Therefore we will restrict our discussion to the three types most commonly used in the oil industry. These are, the buoyancy type, which is the familiar ball-float system; the differential type, which makes use of the difference in head between two columns of liquid; and the displacement type, which uses the torque tube or cantilever spring.

All of these types can give a direct reading on a scale at the location of the primary device, but a much more common use of this instrument has been made by the transmission of the air impulse from an auxiliary pilot, installed at the location of the primary device or float, to a receiving unit located in a central control room.

The ball-float type can be either, externally mounted or be constructed inside the tower. The external type is installed in what is commonly referred to as a kidney, with equalizing lines on the top and bottom leading to the tower. This type definitely limits the effective level area, and this effective area is generally but 8 to 10 inches. It is evident that this type is limited to applications where the level is to be held within a matter of inches. The greatest advantage of the external ball-float type is that when operation of the system is affected because of dirt or damage to the ball, the entire unit can be taken off, inspected and repaired.

The internal type of ball-float system is used where it is desired that the effective area to be greater and a wider sweep is necessary. Although the range of this type is greater than the external type it also has decided disadvantages. The travel of the ball has limitations, and where wide range is desired it is necessary to go to one of the other types. The travel of the internally mounted type depends on the characteristics of the inside of the tower and the length of the extension arm. This system is used where platforms, piping and other obstructions on the outside of the tower prohibits use of the external type. It also is used where the liquid in the tower is at very high temperature and will solidify at the ambient temperature. When used on an application of this kind it eliminates the necessity of steam tracing the float chamber and attached equalizing lines. The use of this type is limited due to the necessity of shutting down the tower when it is necessary to work on the level indicator.

In either the internal or external buoyancy types, the sensitivity depends a great deal on the specific gravity of the liquids being measured. At low specific gravities, there will be little buoyancy to overcome the friction found in the system. Because the size of the float and connecting linkages make it necessary to have a comparatively large rotary shaft and this shaft is necessarily packed to keep it from leaking, the friction ratio is high. The only way to decrease this ratio is to increase the size of the ball float. And this obviously has its physical limits.

The buoyancy types are being used less as popularity of the displacement and differential types increases.

Introduction of the displacement-type liquid-level measuring instrument has filled a vacancy in the instrument field for a long range, compact piece of equipment that has certain advantages of the buoyancy type and still is not restricted to a limited range. The increasing number of applications of this type speaks for itself as to the reception it has received from experienced instrument men.

The principle of the displacement type is based on the weight measurement of displacement. The float is

weighted to sink in the heaviest liquid encountered and is freely suspended from the float arm, which in turn is rigidly connected to a torque tube. The weight of the float will have a downward pull on the float arm and will rotate the torque tube from four to seven degrees. As the liquid in the vessel rises, the float is buoyed up by a force equal to the weight of the liquid displaced. This in turn, releases the tension on the torque tube, causing rotation of the shaft toward normal position.

Among the advantages of the torque-tube liquid-level instrument, is that the distance the float has to travel is relatively small as compared to other types, but it has a wide range of level indication. This movement in a 14-inch float type is only ⅝-inch for complete travel. A choice of ranges from 14 to 120 inches is available. Another advantage is the freedom from extra guides which would normally increase the friction ratio. There is no stuffing box to leak.

The disadvantages of this type are comparable to those of the buoyancy types. As for the exception of the limited rangeability of the ball float, this type has similar general installation characteristics, and problems met with one type generally will occur with the other.

The differential type of liquid-level measuring equipment can be compared with the flow meter, as the mechanism and physical characteristics are practically identical. The chief difference between the conventional flow meter and the differential liquid-level meter is the construction of the mercury-column chamber. In the flow meter the float chamber is installed so as under conditions of no head the mercury is in the usual high-pressure or float chamber, while in the liquid-level meter, under conditions of no head the mercury is in the low-pressure chamber. This is brought about by means of dropping the low-pressure chamber to a point where this condition will exist.

The reason for this change in construction is obvious after consideration of the liquid-level problem. If the conventional differential meter were used and the taps to the meter connected to the tower in the usual manner, the outside pipe leg would be filled with condensate at all times, and consequently a liquid head would be imposed on the mercury chamber. In the usual flow meter this would place the pen at the outside edge of the chart with an empty tower. This method has many disadvantages from the operator's standpoint. But if the range tube is dropped to a point that when the outside leg is filled with condensate the amount of mercury in the low-pressure chamber would be displaced to bring the mercury up in the float chamber, then the pen would be in the usual zero position, and would indicate no level when the pen was near the center of the chart. When using the dropped range tube manometer for liquid level, the pressure taps to the tower are reversed from the usual order.

It may be added, in the electrical induction type of meter, where the transmitting impulse to a receiver is electrical, the conventional meter may be used but the position of the lead wires must be reversed.

One advantage of the differential type is that the pressure taps to the tower are smaller than for the other types. Also, there is a wide level area, which is only limited by the size of the meter and the distance between the pressure taps. The meter mechanism can be taken out of service easily for repair. The temperature, viscosity and other characteristics of the fluid can be ignored, or if necessary, liquid seals can be installed. A record of the level is more easily available without the use of auxiliary systems, and this type eliminates practicaly all friction, with a consequent increase in sensitivity.

Chief among the disadvantages of the differential type is the inherent error due to the difference in temperature and gravity of the liquid leg and the level within the tower. Although this error can be modified where fairly constant conditions prevail, by adding or subtracting mercury where necessary or by making use of the adjusting mechanism found in the meter to restrict or increase the travel of the pen for different mercury levels. This disadvantage has limited use of this type to applications of comparatively stable and uniform conditions.

### Installation of Buoyancy and Displacement Instruments

Installation of the buoyancy and displacement types are somewhat similar. In the external types the float cage must be installed so the center of the cage will correspond with the position of average level. The cage is connected to the tower by means of 1½ or 2-inch pipe. In the larger sizes, the float chamber should be externally braced to the tower to minimize the danger of the connecting nipples breaking.

The valves from the equalizing lines to the tower should be gate valves, as needle valves have a tendency to trap vapors. On the bottom line to the tower, a pipe tee and valve should be installed to facilitate blowing sediment from the float chambers. Careful attention must be paid to the location of the bottom connecting line, for if it is placed in an improper location, sediment may locate in it and plug the equalizing line to the float chamber.

On some applications it may be advisable to install a system to clean out the lines to the chamber in case they get plugged. This can be done by means of installing pipe tees and gate valves in the lines to the tower. A special reamer with a packing gland is inserted into the valve and the valve can then be opened and the line reamed out. All loose sediment can then be washed out. The advantage to this system is that the float chamber will not have to be dismantled from the tower to clean plugged lead lines.

In the internal buoyancy type, it is necessary for the flange to be installed on a sweep type nozzle. This will permit a complete travel of the float. Sometimes, in horizontal towers of small diameter, the ball and arm is installed at right angles to the connecting shaft. This will permit a much larger float range under certain conditions.

In either type, buoyancy or displacement, if the liquid in the tower is in a turbulent state, proper baffles should be furnished to allow the float to operate in a comparatively quiet zone.

Installation of the differential-type liquid-level instrument to a great extent parallels installation of the same type of equipment on flow. The chief difference between the two is the differential-producing device.

There are two ways of producing a differential from a liquid level. The bubble system, in which the differential is conveyed by two columns of gas or air, and no condensate is allowed in the system. One column is above the surface of the liquid and the other below. This type is not extensively used in the oil industry, but with the increased use of catalysts

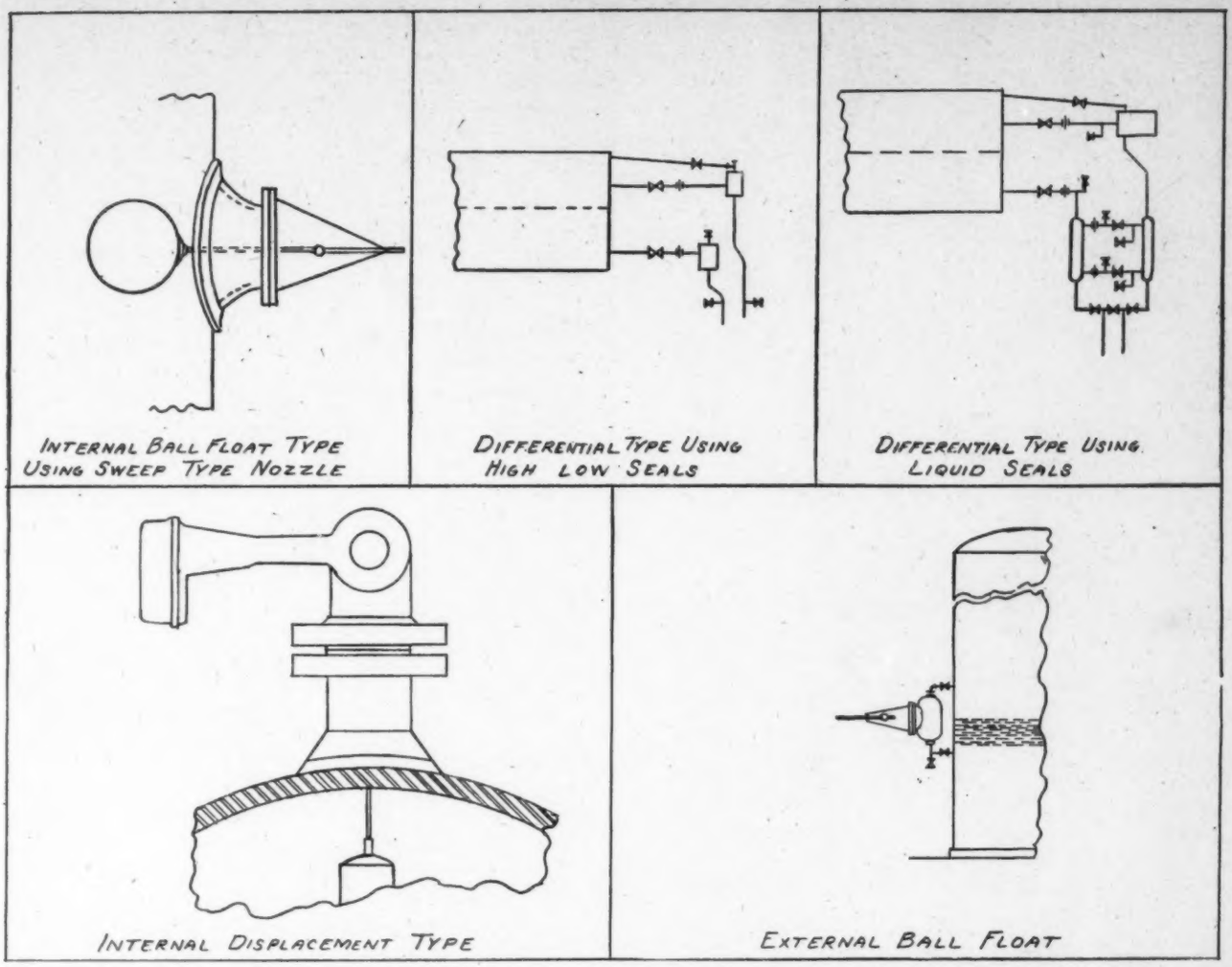

INTERNAL BALL FLOAT TYPE USING SWEEP TYPE NOZZLE
DIFFERENTIAL TYPE USING HIGH LOW SEALS
DIFFERENTIAL TYPE USING LIQUID SEALS
INTERNAL DISPLACEMENT TYPE
EXTERNAL BALL FLOAT

FIGURE 3

and various acids in modern processes more of these applications will be seen in the future.

The second and more common type is the condensing system. This system uses a condensing chamber which serves to automatically maintain a constant liquid head at the point of high-level indication by condensing vapor from the vessel and allowing the excess to flow back into the tower.

Typical installations are shown in the accompanying drawings, Figures 3 and 4. Liquid seals are used whenever the liquid being measured is corrosive, or it will solidify at ambient temperatures or when the liquid is of a volatile nature.

### Sealing Hookups

A choice of sealing hookups is available. In some applications it is preferable to use what is commonly referred to as high-low seals. With this system a seal pot is located at each pressure tap, and the complete lines and meter sealed from contact from the liquid in the tower. This type of installation is preferred where it is desired to keep heavy viscous liquids from the lead lines to the meter.

Another system is used, which in effect, affords protection for the meter body and float mechanism. This will be recognized as being somewhat similar to the liquid seals in the discussion on flow meters. The difference in the liquid-level installation is the addition of the condensing level at the point of high level. This condensing chamber must be large enough to maintain a practically constant level in the chamber regardless of the movement of the mercury in the meter body. The usual dimensions of the condensing chamber is never less than 4 inches in diameter and 6 inches long, and equipped with a filling plug on the top. It should be placed horizontally and be connected to the tower by ½-inch or larger pipe. A ½-inch pipe should lead from the top of the condensing chamber to a tap in the tower to allow uncondensable gasses to return to the tower. If this is not done, the meter will be subject to vapor binding and consequent errors. The return line from the condensing chamber to the tower should not enter the towers more than one tray above the location of the condensing chamber inlet, as at a greater distance a difference in pressure might occur and be of sufficient magnitude to cause a high velocity through the vapor return line.

The purpose of the condensing chamber is to maintain a liquid head at the point of high level by condensing vapors from the tower and allowing the excess to flow back into the tower.

Sealing liquids should be used wherever necessary, but when it is possible the meter and meter piping should be sealed with the tower condensate. Any leak in the seals in liquid-level installations will cause a prohibitive error.

It is generally preferred to keep the specific gravity

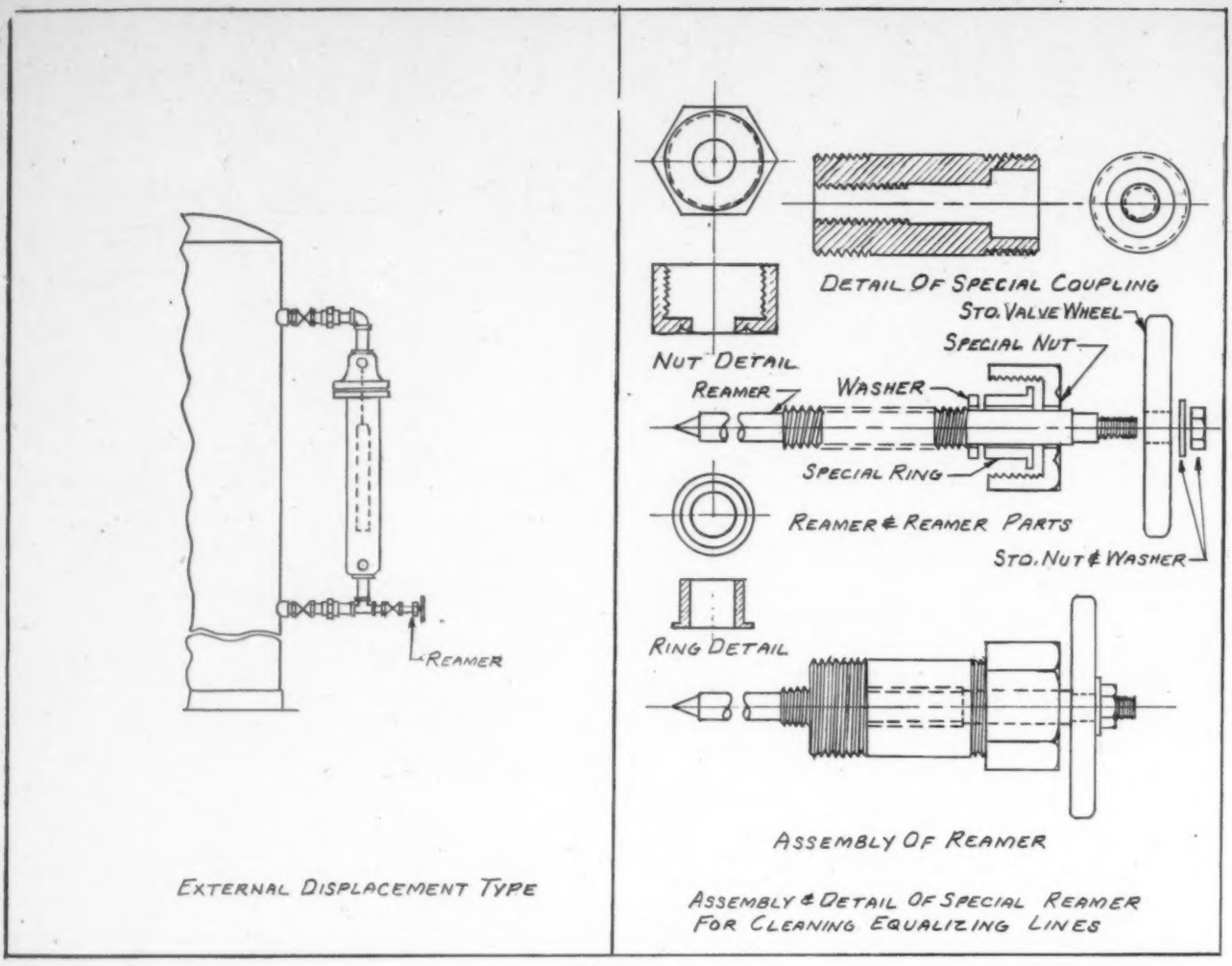



FIGURE 4

ratio between the inner and outer legs no greater than 2 to 1. For example when using a sealing liquid, which has a specific gravity of 1.125, the liquid in the tower should not have a specific gravity of less than 61.

### Maintenance

Maintenance of the buoyancy type and displacement types of instruments will be chiefly periodic inspection and replacement of worn parts and cleaning of the shaft mechanism. Occasionally on some application it will be necessary to ream out the lines into the chamber. Lubrication of the shaft will be necessary in the buoyancy type. With a minimum of attention either of these types will give years of continuous service.

The differential type of installation, especially those equipped with seals, require considerably more maintenance than the other types. But it is possible to keep this type in proper working order for long periods. The chief source of trouble is vaporization of the liquid in the lead lines, but with proper care in the installation of the equipment the error from this source can be held to a minimum. Occasional cleaning of the meter is recommended, especially is it advisable to drain the mercury and dry the meter, as the meter is normally in a low spot and moisture will accumulate and settle in the meter body.

Careful attention should be given the care of the pressure-tight bearing shaft, as the sensitivity of the instrument depends on the friction ratio and this shaft is the greatest source of friction in this meter.

The accuracy of this meter depends on the proper relationship between the inside and outside legs, and the maintenance of these proper conditions give the instrument man his greatest source of trouble. If these conditions are not such that the level in the tower will make full scale travel on the indicator for a complete level change from empty to full, or vice versa, it will be necessary for the operator to make certain mental calculations to know where his level is at any time. Therefore, considerable use should be made of the sensitivity slider in the meter, by which, pen travel to level change can be adjusted.

*The second installment of this discussion of refinery instrumentation will take up temperature and pressure recording and control. It will appear in an early issue.*

# Synthetic Lubricants from Ethylene Condensations

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Technical High School, Dresden

Translated from: Z. Angewandte Chemie, 47 (1934)
No. 39, pp. 677-681
Translated by: E. J. Barth, Petroleum Technologist

**IT CAN be said that by the condensation of ethylene with coal-tar fractions valuable lubricants are obtained in good yields. The viscosity of such synthetic oils can be further increased by treatment with silent electric discharges. These oils show good stability under heat, are free of asphalt and potential sludge bodies, and do not form tars during oxidation at 120° C. They are resistant to oxidation. The synthetic electrically-treated oils still show unsatisfactory and low viscosity index but this disadvantage can be easily overcome by the addition of voltolized fatty oils. Perhaps flat viscosity-temperature curves may be obtained by employing some definitely chosen electrical voltage and tension, or special charges of other frequencies than those we have used in this work.**

IT IS well known that substantially viscous synthetic lubricating oils can be obtained by the hydrogenation of coal tar, and that such oils possess, in many cases, an extremely high viscosity. These oils have not been commonly used to any great extent due to their poor viscosity-temperature characteristics, the presence of potential asphalt-forming agents, and due to their tendency to form tars and sludges during use.[1]

Another procedure for manufacturing synthetic oils from coal tar consists in the condensation of some specific coal-tar fraction with ethylene in the presence of aluminum chloride. The alkylation of naphthalene, for instance, leads to the formation of viscous oils and this procedure has been covered by many patents.[2]

Using another catalyst, C. C. Wulff had shown that anhydrous aluminum chloride could be replaced by boron trifluoride, which is a gas and can be recovered in the process. He found that this catalyst is a very good condensing agent and not so strongly splitting at elevated temperatures as is aluminum chloride. We undertook the experiment described below using naphthalene in order to study the formation of these synthetic oils and to determine their many properties in relation to their mode of formation. We have also employed tetralin and enlarged our work by also using a coal-tar distillate boiling between 200-300° C.

### Preparation of Synthetic Lubricants

The alkylation was conducted in a horizontal high-pressure autoclave of two-liter capacity, equipped with a motor-driven stirrer. Raw materials used in the experiment were the technical grades of naphthalene and tetralin and a crude coal tar which had been previously treated to remove bases and acid bodies. The asphaltic material in the tar was removed by treatment with petroleum ether. The ethylene used contained 96 percent pure olefine. In each run 300 grams of the material were charged into the autoclave with 5 percent anhydrous aluminum chloride after which ethylene was pumped into the apparatus up to an initial pressure of 60 atmospheres. The rate of heating the autoclave was in the neighborhood of 5° C. per minute. The conditions of operation and yields are shown in Table 1. Figure 1 below shows the shape of the viscosity temperature curve for the naphthalene and tetralin oils.

In the case of naphthalene the reaction began at 100° C. and was quite appreciable at 100-110° C. In the course of the next quarter of an hour of continued temperature rise up to 190 the pressure dropped rapidly and steadily down to 5 atmospheres. Upon cooling the autoclave the pressure had diminished steadily to zero. The contents of the autoclave were again treated with a fresh quantity of ethylene to the same initial pressure but it was not until a temperature of 180-190° C. had been reached that there was any noticeable decrease in the pressure. Upon heating to a temperature of 250° C. within the next 45 minutes a noticeable drop in pressure from 105 to 37 atmospheres took place, which pressure finally

**TABLE 1**
**Reactions of Ethylene with Naphthalene, Tetralin and Coal-Tar**

| | Grams Used | Initial Ethylene Pressure (ATM.) | Temp. °C. | Maximum Temp. °C. | Maximum Pressure (ATM.) | Duration of Heating (Hrs.) | Final Pressure at 20° C. (ATM.) | Yield of Crude Product (Gms.) | Remarks |
|---|---|---|---|---|---|---|---|---|---|
| Naphthalene | 300 | 58 | 100 | 190 | 102 | 0.5 | 1 | .... | * |
| | .... | 58 | 180 | 250 | 105 | 2.0 | 1 | 645 | |
| Tetralin | 300 | 49 | 250 | 305 | 122 | 6 | 7 | 436 | |
| Coal-Tar Fraction | 350 | 54 | 290 | 300 | 195 | 6.0 | 40. | .... | * |
| | .... | 58 | 310 | 330 | 210 | 3.0 | 6 | 464 | * |

* Charge of ethylene twice.

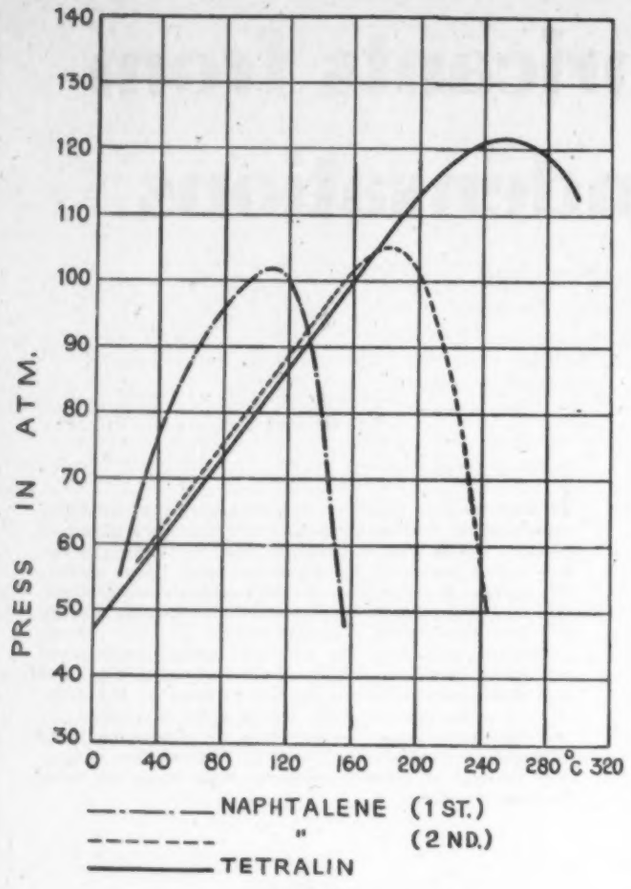


FIGURE 1

Temperature-press. Curve with Ethylene

dropped still lower upon cooling the apparatus. The composition of the gases remaining in the autoclave are:

| | | | |
|---|---|---|---|
| $CO_2$ | 2.0 vol. % | CO | 3.2 vol. % |
| $C_nH_m$ | 47.9 vol. % | $H_2$ | 1.0 vol. % |
| $O_2$ | — | $CH_4$ & higher | 40.0 vol. % |

Tetralin called for a reaction temperature of 250-260° C. When using a total period of 6 hours and up to the higher temperature of 300° C., the final pressure in the apparatus upon cooling was found to be 7 atmospheres. The material was not treated a second time with ethylene. The treatment of the coal-tar distillate was next undertaken. In the first treatment and at a maximum of 300° C. the pressure in the autoclave upon cooling had dropped to 40 atmospheres. The period of heating in this case was 6 hours. The ethylene consumed in this experiment was now replaced, and the pressure dropped rapidly within a short time during the heating up to 330° C. Upon cooling the apparatus at the end of the reaction the pressure was only 6 atmospheres.

### Properties of the Synthetic Lubricants

The reaction products obtained in the above-described runs consisted of dark, reddish oils with strong green cast and which in every case contained considerable hydrogen chloride. The yields obtained using naphthalene and tetralin were close to theoretical, while in the case of the coal-tar the yield was somewhat lower. Besides the liquid products obtained, the reaction also produced a very viscous tarry material which was scraped out of the autoclave. This sludge was an addition-compound of unsaturated oils with aluminum chloride.

The supernatant liquid products were next treated with dilute hydrochloric acid, caustic soda and finally washed free of alkali with water; after drying with calcium chloride the crude reaction product was next distilled at ordinary pressure up to a temperature of 200° C. This was done in order to remove low-boiling constituents up to that point. The material remaining in the distillation flask was next subjected to a vacuum distillation to obtain further fractions as shown in Tables 2-4.

The crude reaction-product obtained from naphthalene furnished lubricating fractions considerably lighter in color and these showed no darkening on standing. Their content of hydrogen was approximately 11 percent. The fluidity of the products decreased with the increase of the boiling point and

**TABLE 2**

**Naphthalene-Ethylene; (Chge: 308 gms.)**

| Fraction No. | Boil. Point °C. | Boil. Point mm. Hg. | Yield Gms. | Yield Percent of Charge | %C | %H | Mol. Wt. | Density 18° C. | Viscosity °E. 50° C. |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 70—200 | 760 | 13.88 | 4.5 | .... | .... | .... | 0.705 | .... |
| 2 | 110—180 | 4. | 44.53 | 14.5 | 89.5 | 10.6 | 221 | 0.954 | .... |
| 3 | 180—186 | 4. | 55.78 | 18.1 | 89.6 | 10.62 | 224 | 0.959 | 6.02 |
| 4 | 186—190 | 4. | 39.82 | 12.9 | 89.5 | 10.7 | 264 | 0.961 | 13.4 |
| 5 | 190—196 | 4. | 60.33 | 19.5 | 89.8 | 10.93 | 273 | 0.962 | 16.5 |
| 6 | 196—210 | 4. | 26.54 | 8.6 | 89.2 | 10.90 | 286 | 0.964 | 35.4 |
| 7 | 210—270 | 4. | 36.06 | 11.7 | 88.5 | 10.97 | 312 | .... | 195.0 |
| Residue | ..... | .... | 17.41 | 5.7 | .... | .... | .... | .... | .... |
| Loss | ..... | .... | 13.65 | 4.5 | .... | .... | .... | .... | .... |

**TABLE 3**

**Tetralin-Ethylene; (Chge: 377.25 gm.)**

| Fraction No. | Boil. Point °C. | Boil. Point At mm. Hg. | Yield Gms. | Yield Percent of Charge | %C. | %H | Mol. Wt. | Viscosity °E. At 20° C. | Density At 18° C. |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 78—230 | 760 | 19.45 | 5.26 | .... | .... | .... | .... | 0.914 |
| 2 | 60—110 | 8.0 | 43.23 | 11.4 | .... | .... | .... | .... | 0.947 |
| 3 | 110—120 | 8.0 | 35.28 | 9.36 | 89.34 | 10.28 | 144 | 1.20 | 0.947 |
| 4 | 120—130 | 8.0 | 40.37 | 10.80 | 89.10 | 10.62 | 168 | 1.26 | 0.948 |
| 5 | 130—135 | 8.0 | 55.12 | 14.20 | 89.22 | 10.48 | 181 | 1.50 | 0.951 |
| 6 | 135—145 | 8.0 | 53.03 | 14.10 | 89.17 | 10.65 | 200 | 2.14 | 0.953 |
| 7 | 145—155 | 8.0 | 61.84 | 16.5 | 88.7 | 11.10 | 210 | 5.45 | 0.957 |
| 8 | 155—240 | 8.0 | 43.78 | 11.70 | 88.72 | 10.92 | 242 | 27.5* | 0.969 |
| Residue | ..... | .... | 17.7 | 4.7 | .... | .... | .... | .... | .... |
| Loss | ..... | .... | 7.45 | 1.98 | .... | .... | .... | .... | .... |

* At 30° C.

**TABLE 4**

**Coal-Tar Dist.-Ethylene: (Chge: 390 gm.)**

| Fraction No. | Boil. Point °C. | Boil. Point mm. Hg. | Yield Gms. | Yield Percent of Charge |
|---|---|---|---|---|
| 1 | 90—125 | 14 | 87.14 | 22.3 |
| 2 | 125—165 | 11 | 101.66 | 26.0 |
| 3 | 165—290 | 11 | 128.10 | 33.0 |
| Residue | ..... | .. | 57.05 | 14.6 |
| Loss | ..... | .. | 16.05 | 4.1 |

**TABLE 5**

**Synthetic Oils with Specifications**

| | Oil From Naphthalene | | Oil From Tar Distillate | Specifications for Motor-Oil |
|---|---|---|---|---|
| | Fraction 3 | Fraction 4 | | |
| Viscosity at 122° F. (°E.) | 6 | 13.4 | 2.0 | 4–28 |
| Flash, open (°C.) | 174 | 186 | 156 | Over 170 |
| Solid-point (°C.) | —9 | —4 | —23 | Over +5 |
| Acid-value | 0.0 | 0.0 | 0.02 | Under 0.3 |
| Percent moisture (%) | 0.0 | 0.0 | 0.0 | Under 0.1 |
| Hard asphalt (%) | 0.0 | 0.0 | 0.0 | Zero |

the molecular weight of the fractions. The viscosity of the last fractions lay in the range of heavy industrial lubricating oil.

According to the percent of carbon and hydrogen in relation to the molecular weight of these fractions it appears that this type of lubricating oil consists of the tetra- and higher-alkylated naphthalenes, as well as dinaphthyl. The viscosity of these oils is surprisingly high in relation to their molecular weight. In the case of petroleum lubricants one is accustomed to deal with molecular weights of 400-500, while in the case of the synthetic oils the most viscous fraction barely showed a value of 300. The same applies also in the case of the large increase in viscosity with only small increases in the molecular weight of these fractions. One may mention at this point the findings of Spilker and Zerbe[3] who found that the number of alkyl groups in the aromatic ring greatly influences the viscosity toward more viscous oils.

The products of the reaction of ethylene and tetralin (see Table 3) were considerably thinner although the viscosity of the individual fractions also increased with increase in the ethyl chains. Only 27 percent of the charge to the distillation flask (the last two fractions showed lubricant properties) were oils which showed lubricant properties. The remainder, or bulk of the distillates consisted mostly of di- and triethyl alkylated tetralin, and these showed viscosities of from 1.2-5.5 E at 20° C. The crude reaction products obtained from the coal tar were vacuum distilled and three fractions were obtained as shown in Table 4. Fraction No. 3 boiling at 165-290° C. at 11 mm. pressure consisted of material of lubricant character and was approximately one third of the crude reaction product. The dark, reddish-yellow oil of 7.4 E. viscosity did not precipitate any solid material and was still liquid at —20° C. The middle fraction consisted of low-viscosity oils similar to a gasoil.

The properties of two oils prepared from naphthalene as compared with the oil from a coal-tar fraction are shown in Table 5. Included in the table are the general specifications for motor oils.

It can be seen that the naphthalene oils in particular pass the specifications fairly well. In the case of the coal-tar lubricant its viscosity and flash-point are below requirements. This can be remedied by a light fractionation and removal of low-boiling-point cuts to produce oils of desired properties. A rather unique property of these synthetic oils is their outstanding low pour-point; these oils are free of potential sludge or tar formers, and when subjected to temperatures far below their solidification points there is no separation of waxes or other solid material.

Figure 2 shows the viscosity-temperature relationsnips of several of these synthetic oils. For comparison we have included a solvent-refined Pennsylvania motor oil, and a light and heavy ordinary commercial lubricant used for motor cars. The Pennsyl-

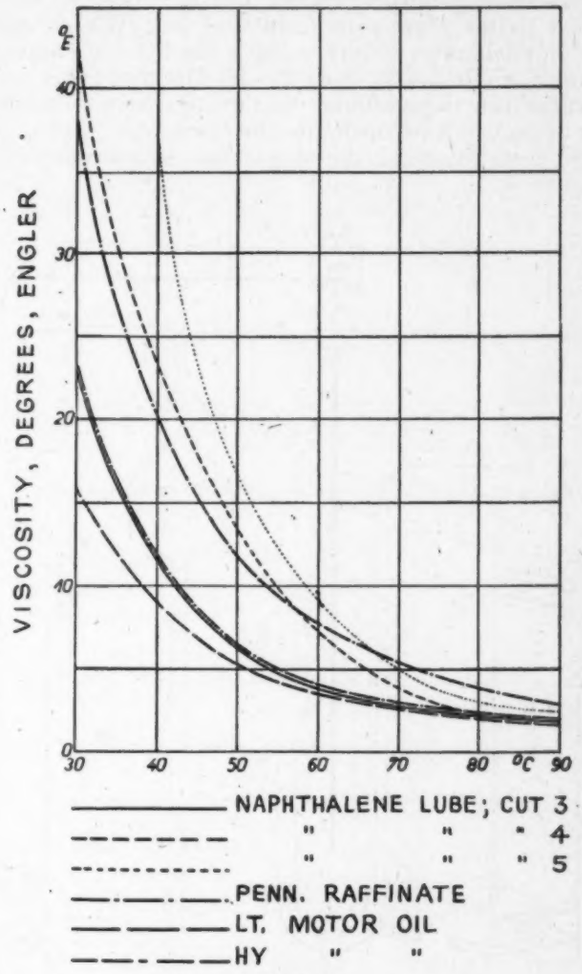


**FIGURE 2**

**Viscosity-Temperature Curve of Various Oils**

vania oil shows practically the same type of curve as fraction No. 3 derived from the naphthalene products. The two miscellaneous motor oils show a somewhat flatter viscosity curve than the synthetic oils in question. If one calculates the viscosity index of these synthetic oils it can be said that the viscosity index is low, which approaches the viscosity index of almost straight aromatic lubricants. The curves become steeper with increase in viscosity.

The synthetic oils are exceedingly stable when attempts are made to oxidize them. Oxidation tests on the naphthalene and tar-oil lubricants were carried out at 120° C. for 70 hours according to methods described in the literature.[5] The oxidation is carried out using oxygen as the oxidizing medium. No sludge or tar was formed, nor could asphaltic material be precipitated by the addition of petroleum ether. The oxidized oils were clearly soluble in the latter. An acid value of 0.09 was obtained in the case of the

naphthalene and the synthetic oil from coal tar showed a value of 0.23 after oxidation.

### The Action of Silent Electric Discharge on Synthetic Oils

We next were interested in determining how our synthetic oils would behave under the influence of silent electric discharges. It is, of course, known that both petroleum oils and fatty oils become very much heavier in viscosity under such influence, and the viscosity-temperature curves of such oils is very much flatter than with untreated oils. We carried out our laboratory work using a modified apparatus similar to that first used by H. Becker.[6] This apparatus functions similar to the pipe arrangements for using ozone according to the Siemans Company.

A description of the apparatus is presented in Figure 3.

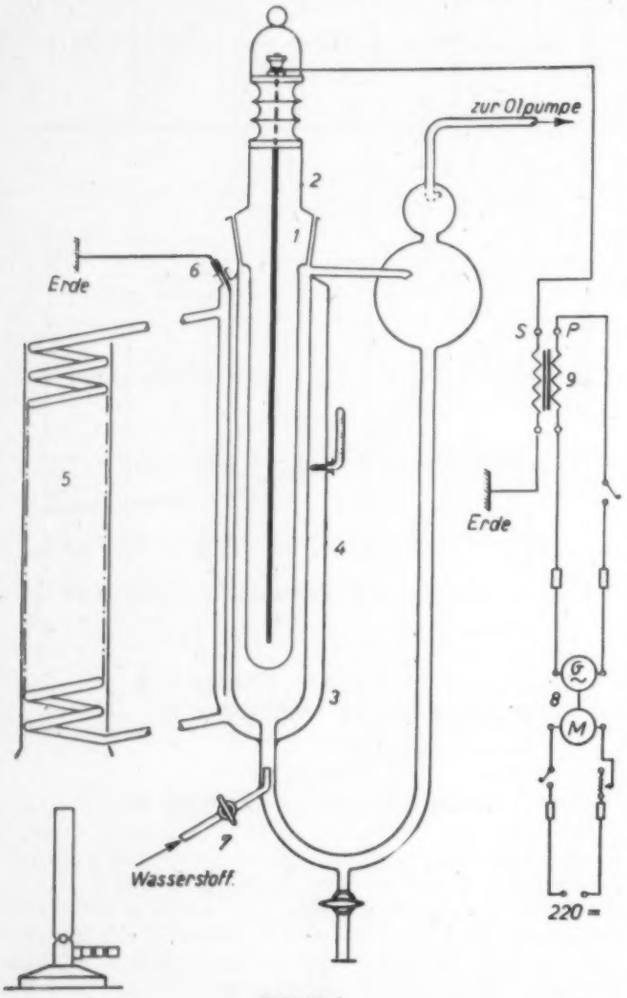


**FIGURE 3**
**Voltolization Apparatus**

The apparatus consists of two glass vessels which fit onto each other by their ground glass joints. The high tension copper electrode **2** enters vessel **1** at the top. This vessel is filled with concentrated sodium bicarbonate solution. The actual high-tension chamber **3** is situated between the heating chamber **4** and the high-tension electrode. The heating-chamber or water-bath situated on the outside of the apparatus is heated by circulating water in the spiral coil heater **5**. The water bath becomes the other electrode, and is grounded by the copper wire **6**. Stopcock **7** is connected to a supply of hydrogen which enters the glass bulb or vessel **3**. When using alternating current of low frequency (we used frequencies with periods of 50 or thereabouts), the electrodes should be spaced only a few millimeters apart, otherwise very high voltages must be employed which are hazardous in glass vessels with thin walls. These precautions are necessary in order to maintain the necessary electrical density during the foaming stage of the operation.

The tension used is 2000 volts and was produced in the electrical generator **8** stepped up by transformer **9**. In carrying out the polymerizations the high-tension chamber was filled a third to a half full with the oil to be used. The water bath is then brought to a temperature of 50-60° C., followed by evacuation of the chamber with an oil pump. Hydrogen is next charged to the apparatus maintaining a pressure of 3 mm. After all air has been displaced from the apparatus and pressures are constant the electrical charge is fed into the vessel. The oil becomes quite frothy and foams almost immediately, acquiring a bluish cast with an apparent lightening of the color. The oil foam escapes into the large bulb at the side of the apparatus, is thus broken, and the resulting oil proceeds to flow back into the bottom of the apparatus.

The oil under polymerization is naturally heavily laden with hydrogen bubbles. Adjacent to where the hydrogen enters and becomes dispersed in the oil as droplets a large concentration of bluish-cast haze or minutely dispersed droplets become evident. The froth appears to consist of droplets surrounded by a tough skin and appear bluish by reflected light. H. Becker[6] has investigated this frothy material in a long series of investigations and found the gas droplets in this froth to consist of molecular hydrogen; the latter is evolved as gas split off during the reaction.

It is not well established as yet whether this splitting proceeds under the influence of bombardment of electrons or whether ultraviolet radiation or thermal effects come into play here. It may well be a combination of all these effects. The tremendous increase in viscosity of the oils treated by silent electric discharge is explained by the assumption that any two molecules having lost their hydrogen atoms by splitting reactions next condense (being unsaturated) with one another to form one heavier and larger molecular-weight molecule. If at this moment the treated oil still contains unsaturated linkages or double bonds then, according to E. Eichwald and H. Vogel,[7] the atomic hydrogen formed by splitting will be found to saturate these double bonds.

In case of our prepared synthetic oils we ended up with very appreciable viscosity increases. The viscosity increases under certain conditions still more but is independent of the time of treatment. The desired objective we had in mind to produce oils with very flat viscosity-temperature curves was not obtained under the conditions chosen for the treatment. We were not able to determine whether the failure to obtain flat viscosity-temperature curves resides in some property of the original oil or whether the use of discharges of alternating current of still higher frequency would have accomplished the desired results.

In Fgures 4 and 5 are shown the form of the viscosity-temperature curves of the synthetic naphtha-

**TABLE 6**
**Properties of Voltolized Synthetic Lubricants**

| Oil From | Voltolization (Hrs.) | Density ISC | Mol. Wt. | Pour Point | Viscosity at 30° C. | Viscosity at 50° C. |
|---|---|---|---|---|---|---|
| Naphtalene | ..... | 0.957 | 232 | —18° C. | 12.5 | 4.0 |
| | 17.0 | 0.962 | 262 | —15° C. | 28.0 | 7.0 |
| Tetralin | ..... | 0.952 | 222 | —24° C. | 7.5 | 2.7 |
| | 13 | 0.964 | 233 | —18° C. | 13.0 | 4.4 |
| Coal-Tar Dist... | ..... | 0.962 | 229 | —23° C. | 4.5 | 2.0 |
| | 7.5 | ..... | ..... | ..... | 6.6 | 2.6 |
| | 13.0 | 0.974 | 250 | —22 | 9.8 | 3.4 |
| | 30.0 | 1.002 | 261 | —14 | 25.8 | 7.2 |

lene and coal-tar oils which were treated by silent electrical discharge for various periods. The differences are greatest for the viscosity at 30° C. and are appreciably greater than the original oil with increase in time of treatment; however, the viscosity

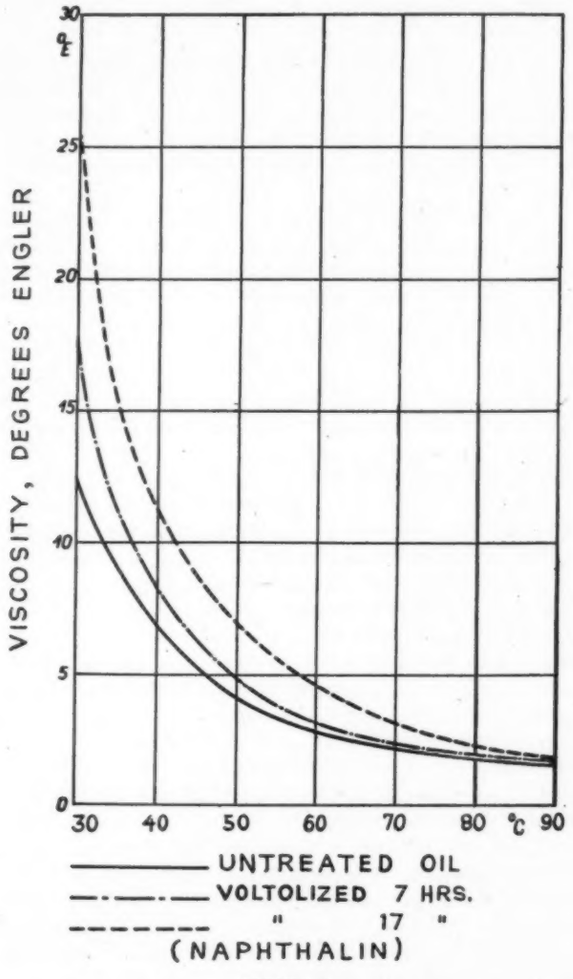


**FIGURE 4**
**Viscosity -Temperature Curve of Voltolized Naphthalene Oil**

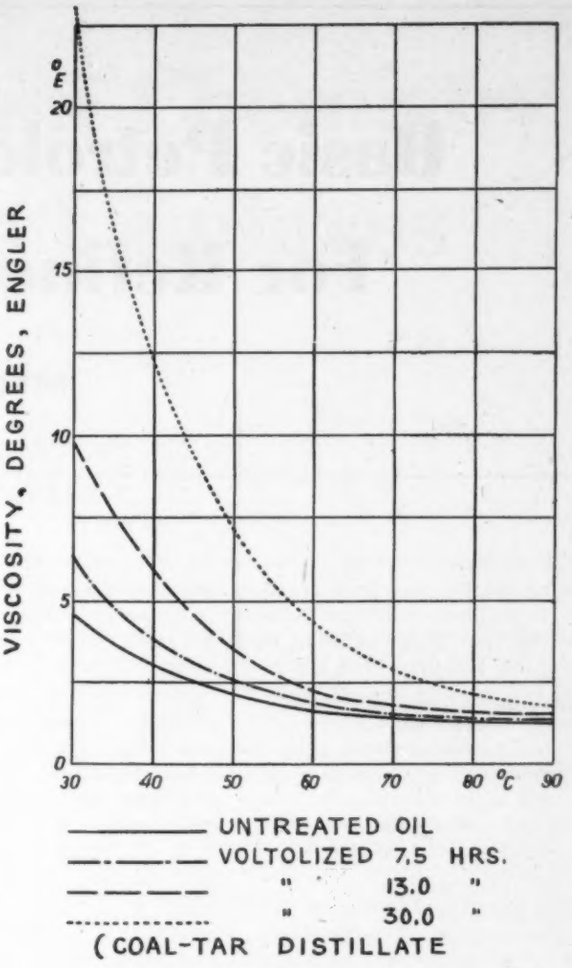


**FIGURE 5**
**Viscosity-Temp. Curve of A Voltolized Coal-Tar Synthetic Lubricant**

at 70° C. drops off sharply, and at 90° C. the value is as low approximately as the original oil before electrical treatment. This is similar to the many petroleum oils found on the market. Despite the large increase in viscosity, the molecular weight has risen relatively little, as is seen by inspection of Table 6.

Low pour point and the excellent stability at very low temperatures of these treated oils should be pointed out despite the extreme viscosity; the pour points are not very much different from the untreated oils.

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[5] Lunge-Berl, IV, 779, Berlin, 1933.
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# Basic Petroleum Chemistry For Refinery Employes

## INTRODUCTION

FOR more than twenty years prior to 1940, the petroleum refining industry had been gradually undergoing a transition into a synthetic chemical industry. A new word "Petrochemical" was coined as being particularly descriptive of this embryonic industry. With the advent of World War II, the transition became sudden, and in a relatively short time refiners were plunged into a gigantic program to synthesize millions of tons of chemical products annually.

Fortunately, much of the technical knowledge necessary for this rapid transition had been developed by chemists, physicists, and engineers over a period of many years. The accumulation of data in the files of industrial-research and educational-institution laboratories spoke volumes in behalf of the foresighted technologists who dared to expend funds on such "impractical" substances as rubber from petroleum and for atomic energy, for instance. Now the results of their efforts in the sudden ending of the war are obvious. Processing units to make synthetic rubber, toluene, cumene, isooctane, neohexane, and many other items of commercial importance, are in commercial production. The embryonic industry has reached maturity almost overnight. With its rapid growth has come an insatiable demand for chemists, physicists, and engineers to man the technical jobs created by expanding industry. Unfortunately, because of the many vicissitudes of war, the limited supply of qualified technologists has caused considerable anxiety among professional and industrial leaders. Even now that hostilities have ceased, the situation is not expected to improve rapidly. The small number of men being educated in the technical professions, as a result of the war, threatens the continued wholesome expansion and development of the new industry into peacetime channels. This is indeed an unfortunate predicament, for because of it, we risk the possibility of losing the world scientific supremacy which we have so recently acquired. We need not lose it, however, if we take full advantage of the talent we now possess. Are we wasting professional talent on jobs where such talent is not required?

That the petroleum industry, in spite of the shortage of technical personnel, has so well succeeded in its war efforts, is due in no small measure to the unselfish efforts of chemist-instructors to produce on the job—not graduate chemists of course—but qualified laboratory technicians. Both men and women, without college training, have been successfully instructed to relieve graduate chemists, physicists, and engineers of routine and non-technical tasks. This has allowed the well-trained scientist to devote his time to important work of an investigational nature. The knowledge and experience of these people are, of course, limited to only the essentials necessary to perform certain tasks. Nevertheless, from this group have emerged improvements in processes and in products. They should be encouraged to pursue scientific knowledge.

The genuine curiosity of these "home-made" technicians has been aroused by the fascinating subject of organic chemistry to which they have had a superficial exposure. The more aggressive now demand at least some knowledge of the fundamentals of chemistry. This is especially true of those men and women who have gone into processing work. They desire to know something about hydrocarbons and what happens to the molecules in the various petrochemical processes such as cracking, polymerization, alkylation, isomerization, aromatization, and others.

Because of the importance of these people to the peacetime activities of the petroleum industry, it should be the duty of technical supervisors to stimulate in operating personnel and laboratory assistants the desire to acquire the fundamental knowledge of chemistry necessary to make them better qualified for intelligent plant operation—and incidentally, happier workers.

The many questions of these people concerning hydrocarbon chemistry have inspired the writing of the following chapters in the language of the refinery worker. It is hoped that in them the reader will find the satisfaction which comes from improving one's knowledge. These writings are intended to supplement the very fine magazine articles which have been appearing from time to time, and which will be specifically referred to in the following chapters. To the technical supervisor, it is hoped this information will be an aid in his duties as foreman-instructor, and an introduction to the many fine books on organic chemistry and petroleum refining technology, a few of which will be specifically referred to in the references.

The petroleum refining industry each year absorbs from the universities and colleges a number of chemists and chemical engineers whose training has been general. Petroleum, during college training, has been to them merely one of the many raw materials which were considered superficially in organic chemistry courses. The specific information contained in this series of chapters on "Basic Petroleum Chemistry for Refinery Employees" should quickly furnish basic and necessary knowledge which might otherwise require much time and experience to acquire.

# Basic Petroleum Chemistry For Refinery Employes

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## Chapter 1
## FUNDAMENTALS

CHEMISTRY is the science of matter and the changes which matter may undergo. Everything which occupies space, everything which we can see, feel, smell, taste, or touch, is matter. Whether it is the unsightly B.S.&W. mess from a slop tank or the beautiful snow-white, crystalline substance which we call salt or sodium chloride, matter is composed of sub-microscopic particles called molecules, and the molecules are subdivided into particles called atoms. For all practical purposes there are about 96 known substances which cannot be further subdivided by chemical means. These are called elements. Four of these elements were discovered as a result of research in connection with the history-making atomic bomb.

Fortunately, of the ninety-six known elements which make up the universe, there are only 2, hydrogen and carbon, which go to make up the bulk of the matter we call petroleum. When these are combined in various proportions they form molecules called hydrocarbons. Petroleum chemistry is basically a study of hydrocarbons.

### Atomic Weight

It is true that other elements such as sulfur, chlorine, oxygen, and nitrogen exist to a small extent in crude petroleum; however, they are relatively unimportant and will be discussed only briefly when we consider treating and corrosion problems.

Since all matter possesses the property we call weight, it follows then that an atom of matter, regardless of size, must have weight. The exact weight of these atoms of matter are of little significance, but their relative weights are very important as we shall see. Because oxygen was one of the earliest elements isolated and because it easily combines with most other elements, it was many years ago chosen as the element upon which a system of atomic weights would be based. In other words, all atoms as they became known were assigned an atomic weight in relation to the weight of oxygen which was arbitrarily assigned the atomic weight of 16.000. For example, it was discovered that helium, a light gas found in gas wells in parts of Kansas and Texas, has an atom which is just about one fourth as heavy as oxygen. It was therefore assigned an atomic weight of 4.003. Similarly, the sulfur atom was found to be slightly more than two times as heavy as oxygen. It was assigned the atomic weight of 32.064.

Early chemists discovered by much experimental data that very definite laws govern the reactions of chemical atoms, molecules, and compounds. A few of the most fundamental of these laws are stated briefly below:

### The Law of Conservation of Mass

When the weight of all substances taking part in a chemical reaction are totaled, it is found that there is no change in the weight of the final mass. That means that if 16 pounds of oxygen reacted completely under proper conditions with 30 pounds of

**THIS series of 10 articles on Basic Petroleum Chemistry has been prepared in order to satisfy the needs of refinery personnel in postwar readjustment. Many men are returning to refinery posts after months or years in military service. The subject matter has been so presented that it will give these men a quick means of regaining familiarity with basic chemistry as it relates to their work.**

**In additon refining plants like other plants are of necessity placing many men in jobs for which they have no basic knowledge. Whatever the work a man does in a refinery it comes in contact with chemical processing. Although he may have no necessity of striving to become a chemical engineer, he will profit by learning something about the nature of refinery processing.**

**It is hoped that the articles will prove helpful in vocational classes and other training programs, which are being expanded because of the postwar conditions outlined above. The ten chapters to be printed in 1946 are:**

**Chapter I, Fundamentals**
**Chapter II, Naming Hydrocarbons**
**Chapter III, Chemical Composition of Crude Petroleum**
**Chapter IV, Changing the Structure of Petroleum Molecules**
**Chapter V, Catalytic Cracking Processes**
**Chapter VI, Manufacture of Aviation Gasoline Components**
**Chapter VII, Petroleum Impurities and Treating Methods**
**Chapter VIII, Petrochemicals**
**Chapter IX, The Refinery of the Future.**
**Chapter X, Potential Sources of Liquid Fuel.**

ethane to produce ethyl alcohol, we would have exactly 46 pounds of alcohol—no more nor no less.

### The Law of Definite Composition

Pure compounds always contain the same proportion by weight of the constituent elements. For example, pure ethyl alcohol will always contain the same percentages of oxygen, carbon, and hydrogen or 34.74 percent oxygen, 52.13 percent carbon, and 13.13 percent hydrogen.

### The Law of Multiple Proportions

If two or more elements yield a series of compounds, the different weights of one which combine with the fixed weights of the other, are in the ratio of small whole numbers. This means, for example, in the cased of a fixed weight of carbon in combination with oxygen, there are two compounds formed —carbon monoxide (CO) and carbon dioxide ($CO_2$). In CO, 12.01 parts of carbon combine with 16.00 parts by weight of oxygen. In $CO_2$, the fixed weight of carbon, 12.01 parts, combine not with 16.00 parts of oxygen, but 32 parts by weight. The ratio is expressed in small whole numbers 16:32 or 1:2.

A more exact or more complete explanation of atomic weights and the atomic theory is not essential for an understanding of basic petroleum chemistry. However, all good books on elementary general chemistry will explain this subject in considerable detail.[1, 2, 3, 4, 5]

The two elements of which petroleum is chiefly composed, hydrogen and carbon, have atomic weights 1.0080 and 12.010, respectively. In other words, hydrogen is about 1/16 as heavy as oxygen and carbon is about ¾ as heavy as oxygen.

With this information, we can easily establish the weight of certain molecules. For instance, everyone is familiar with the chemical formula $H_2O$ which represents a molecule of water.

Two hydrogens + one oxygen = Water
$(2 \times 1.0080) + 16 = 18.0160$

The number 18.0160 is, therefore, the molecular weight of water.

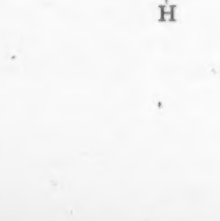
The simplest hydrocarbon with which we have to deal consists of one atom of the element carbon and four atoms of the element hydrogen. It is called "methane" and is represented by the formula $CH_4$.

One carbon + four hydrogens = Methane
$12.01 + (4 \times 1.0080) = 16.0420$

The number 16.0420 is, therefore, the molecular weight of methane.

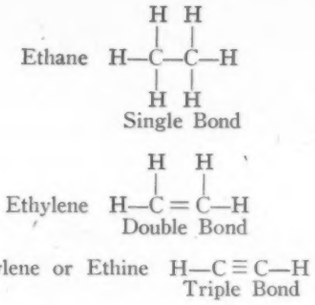
If we were to decompose 18.0160 tons of water into its elements, we would get 16 tons of oxygen and 2.016 tons of hydrogen. These ratios would be the same regardless of the units of weight used—grams, ounces, pounds, or tons.

The formulae $H_2O$ and $CH_4$ are known as empirical formulae. It is important in hydrocarbon chemistry that we also understand the structure of hydrocarbon molecules. The structural formula of water and methane are represented as follows:




It will be observed that the oxygen atom has two "bonds" to which are attached the two hydrogens in water. Similarly, carbon has four bonds to which are attached four hydrogens in the molecule of methane. This will hold true in all hydrocarbon structure, i.e., each oxygen will have two bonds, carbon will have four bonds, and hydrogen will have one. This property is called "valence." Oxygen has a valence of two, carbon a valence of four, and hydrogen a valence of one.

The carbon atom may have single, double, or triple bonds attached to another atom of carbon. For example:




It will be observed also that in each instance above, the carbon atoms have four bonds while the hydrogen atoms have only one.

The hydrocarbons which have only single bonds are known as saturated hydrocarbons or "paraffins." The word paraffin is a derivative of the Latin word meaning "little affinity" or little tendency to react with other substances. The hydrocarbons having double and triple bonds in their structures are known as unsaturated hydrocarbons, and unlike the paraffins they react readily with other substances.

Saturated compounds predominate in the straight-run gasoline and naphtha from most crudes. This will explain why straight-run gasolines are stable to the oxygen stability test, contain little or no gum, and are particularly desirable for aviation gasoline. However, the higher-molecular-weight straight-run gasolines cannot be used in aviation fuel because their octane numbers are too low. In fact, the saturated hydrocarbons containing more than 12 carbon atoms are not even desirable as automotive fuel both because of very low octane and low volatility. The hydrocarbons having 12 or more carbon atoms, however, are useful in kerosine, furnace oil, diesel fuels, waxes, lubricating oil, industrial fuel, asphalt, and cracking stock for gasoline production. However, before we discuss the various fractions of petroleum, we should get an idea of the system of naming hydrocarbon compounds. For this field of chemistry, there is a high powered term, "nomenclature," which chemists use. This will be the subject matter of our next chapter, but we will call it "Naming Hydrocarbons."

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# The Effect of Measurement Dead Time in the Control of Certain Processes

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**THE purpose of this paper is to report on a series of automatic control tests which demonstrate the importance of instantaneous controller mechanism response in self-balancing potentiometer controllers. The automatic control tests were made with both two-position and proportional-reset control. Other tests dealing with the importance of measurement dead zone of the controller are included, noting the relation between dead zone of measurement and dead time of controller response.**

**This paper was delivered as a talk before the American Society of Mechanical Engineers annual meeting, November 26-29, 1945.**

ONE of the most important factors in automatic control of industrial processes is the manner in which the controller mechanism responds to changes in the measured variable. This measuring lag, as it is often called, forms an extensive part of the input-output controller response relationship.

Generally, the factors of measurement dead time and dead zone are neglected in process-control analysis, particularly in mathematical analysis where their inclusion results in complications of a high order. Therefore, it seems desirable to investigate the effect of these factors in automatic control in order to aid in the application of theoretical or empirical data to control problems.

Potentiometer controller mechanisms which incorporate continuous rather than periodic principles of rebalancing provide an opportunity for comparison test of these types of measuring means. Under simulated control conditions it is possible to analyze the dynamic action of the control system and the effectiveness of continuous measurement.

The measuring lag of a temperature controller is composed of two main parts: the lag of the primary element, and the lag of the controller detecting mechanism. It is this second part of the measuring lag, that due to the detecting mechanism, which we wish to study. For purposes of discussion we may classify the many methods of operation of self-balancing potentiometer controllers under two general types: periodic action and continuous action. A periodic-action potentiometer (recording type) is generally identified by a periodic mechanical system for detecting the position of a galvanometer pointer. When an unbalance of voltages is produced by a change in measured temperature, the detecting mechanism determines the amount of unbalance and drives a voltage divider or slide wire to balance against the new value of measured voltage. The periodic-action potentiometer is in wide general use for both measurement and control of process variables.

The continuous-action potentiometer (recording type) is generally identified[1] by the use of continuous, uninterrupted drive means, usually electronic, for obtaining the balance of measured against standard voltages. The use of continuous action in potentiometer balancing eliminates any lag due to a rebalancing mechanism, and avoids periodicity whereby a small dead time may elapse before changes in controlled temperature can be detected.

### Tests With Two-Position Control

In order to determine the effect of dead time in the measuring system, it is desirable to conduct tests of two-position control. By this means all extraneous factors may be eliminated from the tests of automatic control. Processes were selected on the process analog control board[2] by using various combinations of capacitance, resistance, and dead time.

The reaction curves for the processes to be tested are shown in Figures 1 and 2. A reaction curve is determined by setting the valve position and allowing the measured variable to balance. A small sudden movement of the valve to a new fixed position will result in gradual increase of the measured variable to a new balanced value. The resulting time rate of change of the measured variable is the reaction curve. All processes indicate that self-regulation is present. Although the reaction curves are shown for only one direction of change, it may be assumed that the negative change produces a similar curve.

Process No. 1, shown in Figure 1, is essentially a single-capacity process with a time constant of about 6.0 minutes, which is the time required to achieve 63 percent of final value. Process No. 2 is identical in characteristics except that a dead time of 0.1 minutes was introduced between controller action and resultant valve movement.

Process No. 3, shown in Figure 2, is a two-capacity process and thus involves slight transfer lag as can be noted by the slower initial rate of change on the reaction curve. Process No. 4 is identical in characteristics except that a dead time of 0.25 minutes was introduced between controller action and resultant valve movement.

Each process analog was connected, in turn, to the potentiometer controller. Each potentiometer controller, a periodic-action type and a continuous-action type, was fitted with both pneumatic and electric two-position (on-off) control. Pneumatic control was accomplished through a pneumatic diaphragm mech-

anism. The proportional band (throttling range) of the pneumatic "on-off" controllers was 0.6 percent of controller scale. The "on-off" pneumatic controller is not actually a two-position controller but it is proportional controller with a very narrow band. This controller, however, is commonly termed "on-off". Electric control was arranged so that the control contacts directly actuated the energy supply to the proc-

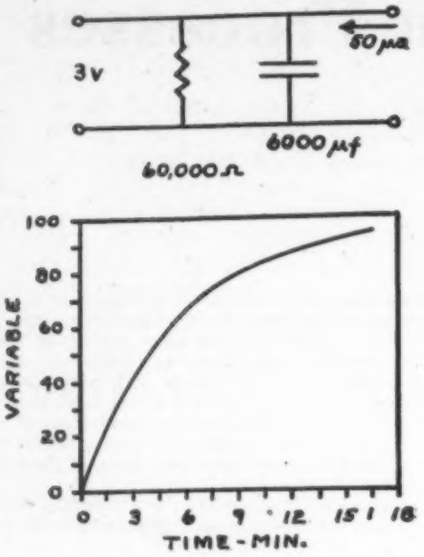


FIGURE 1

ess. The differential gap of both electrical on-off controllers was 0.05 percent of controller scale.

With all process and controller combinations a cycle of controlled variable was recorded. The resulting control cycle is described in Table 1 which shows amplitude and Table 2 which shows period of cycle of the controlled variable.

The results shown in Tables 1 and 2 illustrate the improvement caused by eliminating the dead time delay in the potentiometer measuring means. This effect is particularly noticeable in processes No. 1 and No. 3 where all dead time in the controlled system is concentrated in the measuring means. In processes similar to No. 4 where considerable dead time exists in the process itself, the elimination of measurement dead time results in proportionately less improvement.

Dead time existing in any portion of the controlled system results in nearly proportional increase in amplitude. It is obvious that if automatic control is to maintain an adequate process balance, dead time in the controlled system must be reduced to a minimum.

Applying the results of these tests to applications of automatic control, it is probable that in electrically-heated furnaces and baths where the process lags are very small, the elimination of measurement dead time would result in much closer temperature control.

On the other hand, when controlling the temperature of such processes as heat exchangers where the process lags may be of minutes duration, elimination of measurement dead time may bring no noticeable improvement.

It is interesting to note that the measuring lag may be computed from the results of controlling process No. 1 with the electrical two-position controller. Here, virtually all of the lag aside from the time-constant of the process is associated with the potentiometer measuring means. Since in two-position con-

**TABLE 1**

**Amplitude of Control Cycle—Percent Scale**

| Potentiometer Controller With | Type of Control | Process No. | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 |
| Continuous Action | Pneumatic | .12 | 4.2 | 2.2 | 5.4 |
| Periodic Action | Pneumatic | 1.80 | 4.6 | 4.2 | 7.1 |
| Continuous Action | Electric | .25 | 3.8 | 2.3 | 6.1 |
| Periodic Action | Electric | 1.00 | 4.2 | 4.0 | 6.8 |

**TABLE 2**

**Period of Control Cycle—Minutes**

| Potentiometer Controller With | Type of Control | Process No. | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 |
| Continuous Action | Pneumatic | .10* | .28* | 2.4 | 4.5 |
| Periodic Action | Pneumatic | .50 | .80 | 3.7 | 5.0 |
| Continuous Action | Electric | .03 | .25 | 2.4 | 4.7 |
| Periodic Action | Electric | .27 | .67 | 3.9 | 5.0 |

* Actually throttling to the extent that valve never completely opens or closes.

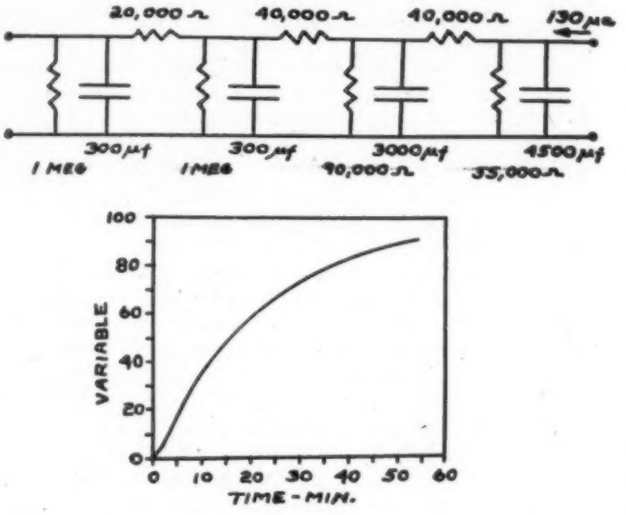


FIGURE 2

trol the period of cycling is about four times the total lag,[3] then the measuring lag should be the period divided by four.

Thus, the measuring lag calculates to be about 0.0075 minutes for the continuous-action potentiometer measuring means and about 0.0645 minutes for the periodic type.

### Tests With Proportional-Reset Control

In order to demonstrate the effect of dead zone in the measuring means, a controller may be applied to a process and the deviation during a recovery from a load change may be expected to show differences depending upon the magnitude of dead zone.[4] The dead zone is generally defined as the greatest range of scale values within which changes in value of the controlled variable are not detected.

With self-balancing potentiometers, the width of dead zone is generally related to the construction of its detecting and balancing mechanism. As one contributing factor, the width of dead zone is inversely proportional to the number of active convolutions of the potentiometer rebalancing slide-wire since the controller pen and controller mechanism is generally positioned to the convolution of the slide-wire nearest the balance point.

The self-balancing continuous-action potentiome-

ter used in the previous tests has 1600 active convolutions on the slide-wire.[1] It was desired to test this same potentiometer with 800 and 400 convolutions on the slide-wire thus multiplying the dead zone by 2 and 4 respectively.

These arrangements were made by altering the mechanical relation between full-scale pen motion and slide-wire contactor travel. An oversize drum on

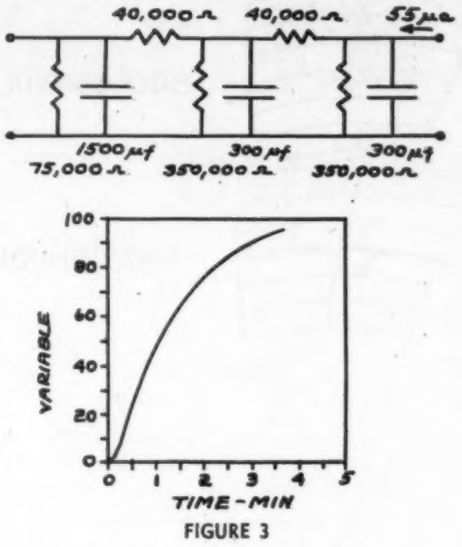


FIGURE 3

the slide-wire driving shaft reduced the amount of slide-wire contactor motion while maintaining the same pen motion. This change does not alter the responsiveness of the potentiometer balancing motor to a given unbalanced emf, but reduces the amount of slide-wire contactor travel for the same pen travel when a rebalancing action occurs. The electronic voltmeter on the process analog control board was then readjusted to maintain the same range of 0 to 5 volts for full-scale pen travel.

The net result of these changes is to alter the number of slide-wire convolutions corresponding to a given pen travel while maintaining the same dynamic balancing action and the same scale calibration.

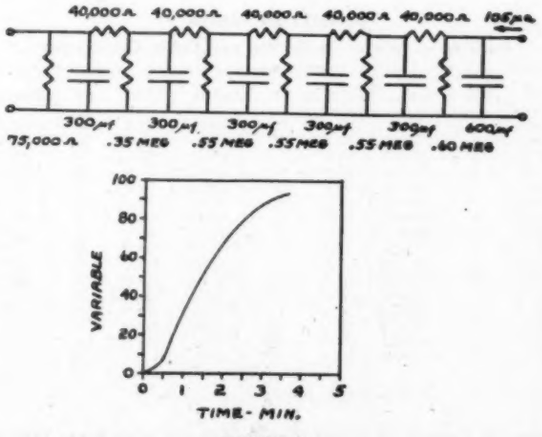


FIGURE 4

Three processes were selected and are described in Figures Nos. 3, 4, and 5. Process No. 5, described in Figue 3, is a relatively simple process having a moderate reaction rate and slight transfer lag. Process No. 6, described in Figure 4, possesses a moderate reaction rate and appreciable transfer lag. Process No. 7, described in Figure 5, has a fast reaction rate and slight transfer lag. In all tests only the dead time inherent in the system was present and no dead time was intentionally added.

Each process analog was connected to the potentiometer controller and optimum adjustments of proportional band and reset rate selected.[5] A pneumatic proportional-reset type controller was employed for these tests. After the controlled variable had stabilized, a change in supply of energy to the process was made and the controller recovery curve obtained.

The supply change was introduced by adding a bias voltage to the existing voltage supplied to the current input unit on the process analog control board. This is, in effect, a change of flow through the control valve caused by a variation in pressure drop and requires that the controller correct for this change. The control valve has 200 effective positions.

The charts showing the recovery curves are reproduced in Figures 6, 7 and 8. The results of these tests are shown in Table 3.

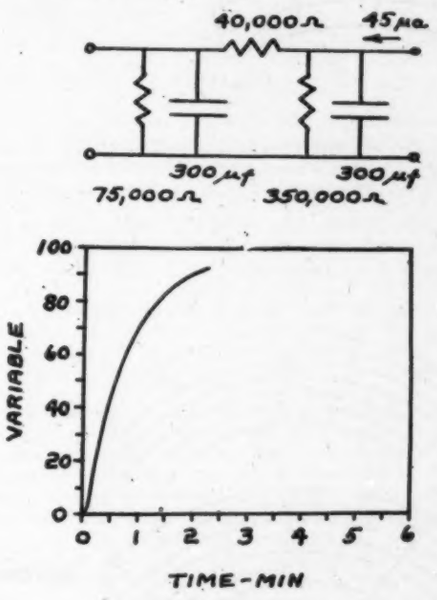


FIGURE 5

As shown by the recovery curves of Figures 6, 7 and 8, both an increase and a decrease in supply was made. The increase in supply results in a rise of the controlled variable before it is corrected by the controller, and a decrease in supply results in a fall of the controlled variable before it is corrected. The maximum deviation was found by adding both the positive deviation and the negative deviation. In this manner, various small non-linearities in valve characteristics, process time-constant and process lag may be averaged.

The difference between the various maximum deviations from a supply change may be attributed to the different widths of measurement dead zone in each test. With a larger dead zone, a short period must elapse between the time when an actual change in measured variable begins and the time when the controller senses the change. Thus, a measurement dead zone creates dead time. This delays the controller corrective action and allows greater deviation.

The stability of control appears to be more cyclic when the dead zone is greater. With Process No. 6 particularly, the recovery curve shows that the proportional band should be slightly increased in order

**TABLE 3**

| Process No. | Prop Band Percent | Reset Rate Per Min. | Max. Deviation for ± Supply Changes No. of Effective Convolutions | | |
|---|---|---|---|---|---|
| | | | 1600 | 800 | 400 |
| 5 | 45 | 0.8 | 11.2 | 11.7 | ..... |
| 6 | 60 | 0.4 | 16.7 | 17.7 | 18.1 |
| 7 | 25 | 3.0 | 10.0 | 10.4 | 10.6 |

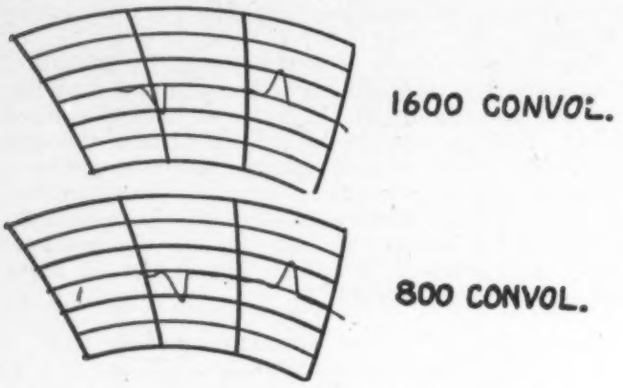


FIGURE 6

to maintain the same ratio of succeeding amplitudes of cycling. There is an appreciably consistent lengthening of period when the dead zone is greater. The apparent decrease in stability of control and the lengthening of period of control points to the conclusion that an additional lag exists when the measuring means of the controller possesses a finite dead zone.

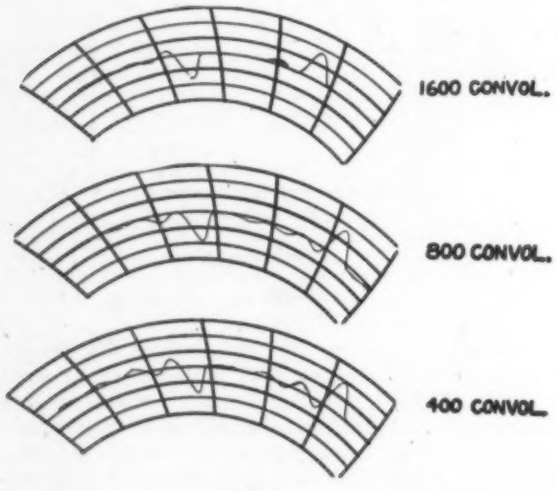


FIGURE 7

## Conclusion

The brief test results given here illustrate the importance of measuring lag and measurement dead zone in automatic control. It is desirable to maintain measurement dead time as small as possible since it increases the quality of control, especially on such processes where all other lags are small. It is desirable to maintain measurement dead zone as small as possible since it increases the quality of control by accomplishing a reduction of dead time.

It is evident that more investigation is required to establish further the relationship between each component part of measuring lag and the measurement dead zone. In most control problems the effect of these factors cannot be ignored because of their influence on the quality of automatic control.

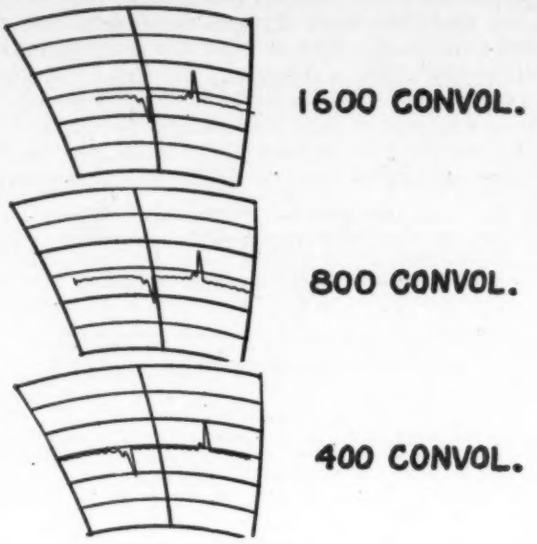


FIGURE 8

The author gratefully acknowledges the suggestions of Mr. T. R. Harrison in the investigation.

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**DENSITY OF WATER**

**Density of Water in Grams per Cubic Centimeter**

| t°F | t°C | Density | t°F | t°C | Density | t°F | t°C | Density |
|---|---|---|---|---|---|---|---|---|
| 32.0 | 0 | 0.999867 | 60.8 | 16 | 0.998969 | 89.6 | 32 | 0.995052 |
| 33.8 | 1 | 0.999026 | 62.6 | 17 | 0.998801 | 91.4 | 33 | 0.994728 |
| 35.6 | 2 | 0.999968 | 64.4 | 18 | 0.998621 | 93.2 | 34 | 0.994397 |
| 37.4 | 3 | 0.999992 | 66.2 | 19 | 0.998430 | 95.0 | 35 | 0.994058 |
| 39.2 | 4 | 1.000000 | 68.0 | 20 | 0.998229 | 96.8 | 36 | 0.993711 |
| 41.0 | 5 | 0.999992 | 69.8 | 21 | 0.998017 | 98.6 | 37 | 0.993356 |
| 42.8 | 6 | 0.999968 | 71.6 | 22 | 0.997795 | 100.4 | 38 | 0.992993 |
| 44.6 | 7 | 0.999929 | 73.4 | 23 | 0.997563 | 102.2 | 39 | 0.992622 |
| 46.4 | 8 | 0.999876 | 75.2 | 24 | 0.997321 | 104.0 | 40 | 0.992244 |
| 48.2 | 9 | 0.999808 | 77.0 | 25 | 0.997069 | 122.0 | 50 | 0.98804 |
| 50.0 | 10 | 0.999727 | 78.8 | 26 | 0.996808 | 140.0 | 60 | 0.98321 |
| 51.8 | 11 | 0.999632 | 80.6 | 27 | 0.996538 | 158.0 | 70 | 0.97778 |
| 53.6 | 12 | 0.999524 | 82.4 | 28 | 0.996258 | 176.0 | 80 | 0.97180 |
| 55.4 | 13 | 0.999404 | 84.2 | 29 | 0.995969 | 194.0 | 90 | 0.96531 |
| 57.2 | 14 | 0.999271 | 86.0 | 30 | 0.995672 | 212.0 | 100 | 0.95835 |
| 59.0 | 15 | 0.999126 | 87.8 | 31 | 0.995366 | 302.0 | 150 | 0.9173 |
| | | | | | | 392.0 | 200 | 0.8628 |

**Apparent Density of water Weighed in Air with Brass Weights (Grams per Cubic Centimeter)**

| t°F | t°C | Density | t°F | t°C | Density | t°F | t°C | Density |
|---|---|---|---|---|---|---|---|---|
| 59.0 | 15 | 0.99805 | 68.0 | 20 | 0.99718 | 77.0 | 25 | 0.99604 |
| 60.8 | 16 | 0.99790 | 69.8 | 21 | 0.99697 | 78.8 | 26 | 0.99579 |
| 62.6 | 17 | 0.99774 | 71.6 | 22 | 0.99676 | 80.6 | 27 | 0.99552 |
| 64.4 | 18 | 0.99756 | 73.4 | 23 | 0.99653 | 82.4 | 28 | 0.99524 |
| 66.2 | 19 | 0.99738 | 75.2 | 24 | 0.99629 | 84.2 | 29 | 0.99496 |
| | | | | | | 86.0 | 30 | 0.99466 |

## SURFACE AREA OF POWDERS

# Heat of Wetting of Metal Surfaces by Oils

H. H. HERBST

*Translated from Kolloid Ziet. 71, No. 3 (1935), by E. J. Barth, Petroleum Technologist*

**THE heat-of-wetting of oils on metallic surfaces is proportional to the lubricity of the former. The heat rise is very small when measured on a straight metal surface. Therefore, the method for heat-of-wetting has been to employ finely divided metallic powders. The disadvantage here, however, is that the true surface area is not well defined when testing out various metals and comparing them with a standard lubricant. In order to determine the surface area one made use of adsorption procedures and assumed that the quantity adsorbed on a smooth metallic bearing is related to the quantity adsorbed by the powder (of the same metal) in the same ratio as bearing surface area to powder surface area.**

**We have now shown that this assumption does not hold for all adsorbents, absorbates and various fine powders. Capillary structure and "ultraporosity" exert considerable influence on these phenomena. For instance, various adsorbates of variable large molecular sizes will tend to give different surface area relationships with one and the same powder.**

**The author, therefore, proposes that the heat-of-wetting of metals by oils be carried out directly on the smooth metal or bearing-metal itself, and describes an apparatus to determine the heat-of-wetting of metals by various oils, colloids, etc.**

A STUDY has been made of determining the physicochemical properties of the surfaces of solid materials. Included in this are the conditions which exist at the interface when a powdered solid is in contact with liquids. It is often difficult to determine the properties of such systems, as for instance, the heat-of-wetting of solids where small units are involved. Such systems comprise not only powdered materials; most often it is desirable to determine the heat-of-wetting and similar characteristics of smooth metallic surfaces by water, oils, lubricants, emulsions, and the like. The physical effect of contact of fluids on solid surfaces is small, and accurate measurements of wetting-ability usually has been difficult, if not impossible, to carry out. One usually did not attempt, for example, to determine the heat rise occurring at the surfaces when a lubricant contacts smooth journals and bearings, or wherever a polar or non-polar substance came in contact with smooth metallic surfaces. The whole field of lubrication is exactly dependent on how and to what extent oils do wet metallic or other surfaces. The whole subject of wetting is quite fundamental also in many other applications in industrial technology.[1,2] One therefore found it more feasible to determine this constant so as to obtain it as a larger measurable unit—namely, to employ the metal in a very finely powdered form or even as a colloid, and to test out various oils for their wetting-ability with such powders. This is based on the assumption that the heat evolved is dependent on the fineness or surface area of the powder in contact with a liquid; that by making the metallic surface eventually to be lubricated of exceedingly large surface area (as by powdering) the evolution of heat is practically proportional then to the surface area, and sufficiently large to be easily measurable.

In doing this one assumes that with increasing heat-of-wetting the greater is the absorption power of oil for the solid. The absorption forces are related in direct proportion to oiliness, or lubricity, or to the lubricating value of an oil. Heat-of-wetting therefore runs parallel to high lubrication value. Lubricity then is dependent on the degree of the forces with which the oil is adsorbed and bound onto the surface to be lubricated. The larger this force, the thinner will be the oil-film before rupturing.

Now it is thus known that metals may be powdered to produce exceedingly high surface-area materials, and a larger heat rise can be obtained than can be measured on a smooth bulk solid surface. One can then test out various oils for lubricity by using a standard finely powdered metal, and noting the heat rise in each case. The standard powdered metal would have to be well and uniformly prepared.

However, there exists an inherent error or difficulty. These difficulties cannot be well eliminated working with some standard powder. The results are ordinarily not translatable into the actual state of conditions existing on the surface of a smooth metallic bearing surface. This is due to:

(1) One usually cannot easily determine or know the actual surface area of metallic powders or materials such as powdered graphite, and

(2) The relative weight or mass unit relationships are very different between oils and fine powders and oils and a smooth metallic surface; in a bearing there exists a thin oil-layer or film in a "metallic field," where quantity of metal far exceeds the amount of oil. The internal molecular forces of the metal are well inhibited or mutually neutralized and only at the interface or surface do they enter the absorbtion phenomenon by residual valency forces. In the case of fine powders or colloidal metals, however, the oil predominates and the metallic substance is present in an "oil-field."

The practice until recently has been to use 250-500 cc. of oil, or 70-150 times more oil than volume of metal, when determining heat-of-wetting; this is in large contrast to the conditions in a bearing, where the volume of oil is only about one one-thousandth of the metal volume—a very large reversal of conditions. The concentration relationships are therefore in both cases some 100,000 times the reverse. In the case of the powders, the oil phase predominates and the fine particles of metal behave more like molecules or molecular complexes. Furthermore, the rate of adsorption is greatly increased by using progressively finer powders and therefore also the amount of heat evolved in unit time. Rate of adsorption, for instance, increases very rapidly with increase in particle size in the case of dense carbons, so that when working with lump walnut-shell carbon ground to a fine powder, one notices at least a 5-10 fold increase in adsorption rate.

With increased speed of adsorption we therefore will measure a relatively greater evolution of heat in a definite period of time. This allows under certain conditions the substitution of a lesser amount of the finer powder as against a metallic powder which is very dense, and which adsorbs slowly. The latter would in unit time evolve only small increments of heat

in comparison. There is then the possibility that in the latter case the heat evolved would be so small in unit time that the calorimeter apparatus itself would be insensitive to recording the rise, and only exceedingly small temperature increases would be evident.

As long as the powder is still relatively coarse-grained, one can determine perhaps a definite proportionality between the ordinary surface-area and adsorption, and therefore the heat of wetting. However, in the case of very fine powders (as those similar to metallic hydroxides reduced by hydrogen after precipitation from solutions of their salts) one must be careful to remember that adsorption phenomena are not as simple as would at first appear. Work on adsorption charcoals has taught this caution; a whole series of observations must be made obtaining data and points on graphs and eliminating effects of decreased heat-rise due to "ultraporosity."[3]

In order to determine the surface area of an active carbon or metallic powder as it is related to the surface of an ordinary smooth metallic system (such as a bearing metal), one makes use of adsorption, and the principle that: quantity adsorbed from the flat metallic surface (i.e. methylene blue dye) is to the quantity adsorbed by the powder as the surface area of the flat or bearing surface is to the surface area of the powder:

$$\frac{\text{adsorbed material on bearing metal}}{\text{material adsorbed by powder}} = \frac{\text{surface area of bearing}}{\text{surface area of powder}}$$

The adsorption of gases, vapors by fluids such as oils, salt-solutions, colloids, radio-active salts, etc., follow this rule, and the adsorbed material can thus be determined. Gases can, for instance, be determined by the use of pressure apparatus, by direct weighing, or by the gas interferometer. In the case of liquids the adsorbed material is determined by titration-methods, colorimetry, conductivity, or electrolysis methods.

The Zeiss interferometer lends itself admirably to measurements of changes in concentration of solutions.

In selecting the above equation as the basis of adsorption measurements, the next step is to determine whether the different adsorbents yield the same absorption-surface area relationships. As will be shown later, this need not always be the case. Only if one were desirous to prove that the related sequence of adsorbed material (in testing their absorption capacity) against various absorbents remains constant would the method be sufficiently practical for testing oil mixtures.

The testing of all kinds of metallic powders for adsorbtion ability naturally would be very significant also for many other industrial applications.

The assumption made to date is that adsorption is entirely a property of the surface area or the function of a material to develop a large surface area, and that by increasing the surface area by decreasing particle size of a material in relation to surface area increase, one could arbitrarily increase the adsorption capacity. These views hardly stand rigorous scrutiny today in view of the experimental findings obtained recently with active charcoals.[3]

Let us investigate, for example, an active carbon, silicagel or similar material. The absorbent may show a very highly desirable large surface-area, but it need not by any means be a very excellent absorbtion medium. It is not so much a case entirely of high surface area when dealing with this phenomenon but very much more a matter of what the interface of the adsorbent consists of, what type of molecules are present there, and are the latter "free" to attract, say by double bonds, or are the molecules saturated, and so forth.

One could at best determine only the active layer at the interface or surface rather than the whole surface itself. The term "interface at a surface" or surface area of a fine powder or colloid need either be revised or else not really employed in discussing the inner mechanism of adsorption; when speaking of activated molecules or aggregates one can no longer refer to them as having a surface area or interface, as is used in adsorption techniques. The molecule comes in the picture of adsorption, not as a surface, but as a spatial configuration or three-dimensional structure.

Let us, for instance, secure a previously treated, purified, tar-free, lumpy and active "coal-carbon," and grind it continuously finer and finer to an impalpable powder. Testing for adsorption we will find that by doing this we obtained no greater degree or amount of adsorption but merely increased the rate of adsorption. H. Fischer, working with an active carbon of 1.25-2.5 mm. particle size diameter, obtained an absorption capacity of 0.552 m mols/gm of succinic acid per gram of carbon at the end of 30 minutes (from aqueous solution), and after 4320 minutes 1.183 m mols/gm of succinic acid. The material of above particle size was next powdered as fine as possible in rotating mortars, and repeating the experiment with this powder, he found the adsorbed material to be 0.935 m mol/gm of succinic acid at 30 minutes, and 1.183 m mol/gm at 4320 minutes. It is apparent from this simple test that by powdering and increasing the "surface" very much, the quantity of adsorbed material was not altered; and that such pulverization had merely increased the rate of adsorption and at that, only in the first interval of time.

Very dense carbons such as those from walnut shells behave differently. Using lumps the size of small peas, this carbon shows an adsorption of 0.057 m mols/gm of succinic acid after 70 hours, and after 336 hours, an adsorption efficiency of 0.099 m mol/gm. If one powders this carbon as fine as possible we obtain at the end of 30 hours ad adsorption of 0.34 m mol/g and at the end of 360 hours 0.839 m mol/g of succinic acid. In this case the activity is increased by pulverization.

In determining the ease or efficiency of adsorption and in consequence the heat-of-wetting of an active substance such as activated carbon, one must, in addition, take into account the property of "ultra-porosity."

Adsorption decline due to lessened ultraporosity[4] is the phenomenon responsible for smaller activity where the adsorbed molecules are larger (as in the case of testing very dense carbons) than when testing out smaller adsorbable molecules. The mechanism is explained as follows: smaller molecules ready for adsorption come in contact sooner and contact more readily the heavily or closely-packed carbon molecule, while the larger adsorbable molecules due to a type of ultrafiltration cannot attach themselves to the active molecules very easily since the capillary entrances or pores are too narrow. One will obtain, therefore, in such cases entirely different results when testing the adsorption-capacity and the heat-of-wetting of materials with a variety of large absorbable molecules; and therefore, as we have shown above by the surface area equation, very different results due to various "surface areas" for one and the same active powder.

We see, therefore, that the adsorption-method is not entirely without its drawbacks, and conclusions should only be drawn with special reservations. One cannot by this procedure measure adsorption-ability of surfaces without additional considerations. It is especially possible that the large "methylene blue" molecule is entirely too big and cannot contact all of the active molecules present, toward which, for instance, a very fluid oil would easily migrate. Testing therefore with methylene blue, one cannot evaluate a reactive surface which would be available to a smaller-molecular-weight substance.

Furthermore, we lack a complete set of adsorption constants of methylene blue toward various adsorption materials, and also for various metals. In adsorption it is not a matter of surface equals surface physically, but what is most important is: what is the chemical nature of the surface. Adsorption constants for various substances are quite different. It seems, however, that methylene blue is a typical substance to show how the phenomenon of ultra-filtration takes place in a carbon or active coal-substance.[5]

At this point I would like to refer to the work of O. Ruff, P. Mautner and F. Ebert entitled "Amorphous Carbon or Graphite." (Zeit. inorg. Gen. Chem. 167, 185 (1927); referred to in Koll. Zeit. 44, 86 (1928). According to Ruff the possibility of adsorption of graphite powders of varying particle size increases with an increase of surface but does not reveal or comprise any increase in adsorption due to greater surface area. For instance, increasing the surface area from 2-50 fold the adsorption of phenol is increased 3-4 fold, and congo-red only 2-8 fold. Activated atoms do not increase correspondingly with the increase in total surface area.

According to this, one would find different surface-area relationships for the same powder when using adsorption methods, as a measure for surface area, as Bachman and Brieger had done (see Footnote 1); It depends on what one chooses as the adsorbable substance for such tests. The use of adsorption-methods to determine surface area is therefore open to criticism and care must be exercised in formulating the conclusions derived therefrom.

As against the above considerations, adsorption-methods and heat-of-wetting do yield valuable information when rightly directed, and are especially applicable when used in conjunction with studies of lube oils and lubrication. I will discuss this further toward the end of this paper.

Using adsorption methods as a measure of "surface interface" we find it does not actually give a comparison or a true picture of all surfaces involved but only a comparison or state of the apparently active surface layers.

The work of Prof. Marc at Jena on adsorption phenomena on crystals in 1913 recalls to mind that Marc pointed out that it is of prime importance to know what the constituents of the adsorbent are. This investigator, reporting in "Concerning

Adsorption and Saturated Surface Areas," (Zeit, physik. Chem. 81, 641 (1912), publishes a series of corollaries and conditions which occur and are necessary for adsorption on crystals.

For example, it was shown that colloids, especially, are strongly adsorbed by crystals, while crystalloids are, in general, much less strongly adsorbed. The adsorption of crystalloids depends essentially upon the form of crystal, etc., and upon whether they can develop solid solutions, etc. Reviewing the table on page 649, there is shown above all, that one cannot draw conclusions from adsorption phenomena as to the size or dimensions of the surface area or interface of crystalline powders when using various crystals as the adsorbent, and working with many types of adsorbable substances. One will perceive instead that adsorption capacity is not at all proportional to the various types of adsorbable substances. The latter do not seem to follow any rule as to the series of substance this investigator had before him.

The same results or conclusions may be expected in the field of metallic powders where we also deal mostly with the crystalline state. The circumstances and relationships are here perhaps even more complicated since we deal here with alloys (of which bearings are mostly composed) which consist of various conglomerates—themselves again composed of various crystalline bodies. The study of adsorption here in the case of such substances should be thoroughly and more searchingly investigated. Above all, it should be established whether there really are differences in the adsorption of crystalloids and colloids. Do the oils follow the rules of adsorption in relation to various adsorbent metals, and is it again quite different when the latter are finely pulverized?

One has in times past also made use of the method of determining relationships between solubility of the solid surface and the powder in order to determine the surface area of the metallic powder. But this method is also not any too reliable for accurate results and is even more difficult to carry out experimentally than adsorption-measurements; for reasons due to the disturbing influences of solubility effects, diffusion and electrical phenomena, and the like. The procedure will not be further discussed. Precipitation methods will not be discussed for the present.

It seemed to the author, from a study of past literature and previous methods that in order to avoid the uncertainties and many discrepancies inherent in surface-area measurements, it was best to determine the small heat-of-wetting directly on the smooth metallic surfaces, even though the heat units evolved would be quite small and of a low order, and from all sides at times apparently negligible. Here at least we come nearest to all the conditions and elements as exist in a bearing, and many of the usual uncertainties in determinations would be by-passed.

Therefore, we stress at once that it is not sufficient to test only the bearing-metal alone (as powder) and disregard the journal metal. One must determine the heat-of-wetting of each separately and again when both are combined and mixed.

For the type of apparatus that was employed in our investigation we are indebted to the firm of Hanff & Buest, who built the special calorimeter with our help in its design.

We found that the usual type of calorimeter used for heat-of-wetting was unsuitable for our purpose because the large amount of oil used has a tendency to absorb the heat evolved; these calorimeters require the use of 250-500 cc of oil used to set the metallic powder. We therefore designed our new calorimeter to operate with a minimum amount of oil and with a small "water equivalent" figure; by this means the minute amount of heat evolved in wetting the bare glass wall, while negligible, is measured and can be used in the calculations.

### Description of Apparatus to Determine Heat of Wetting

The apparatus was so constructed that the heat-of-wetting could be determined on metal wire, turnings, metallic capillary tubing, ribbon, slit cylinders, as well as metallic powders. Any of these metallic pieces are so cut and selected that their surfaces can be accurately measured.

The apparatus to make these determinations is shown in Figure 1. To make the diagram clear, the drawing shown has been drawn slightly larger than the actual size.

The apparatus consists of a vertical upright adsorption vessel, a, of relatively narrow diameter. b is the adsorption space or area containing test-specimen, c, which can be a metal rod, ribbon or the metallic powder; d is an electric resistance thermometer, such as made from thin platinum wire with electrode lead wires $d_1$ and $d_2$; the latter connect to an electrical pyrometer box which may be an arrangement of wheatstone precision bridge, and a very sensitive galvanometer, plus a source of current such as a battery or direct current connection. The resistance thermometer is wound around the adsorption space and may be enclosed in a thin glass casing e, itself filled with hydrogen—or it can be soldered directly onto the adsorption-vessel or space, taking care to insure no short-circuiting or grounding; (to avoid the latter the wires should be wound on very thinly spaced strips of mica or the like). F is the elctrical standardization heating arrangement—such as we used—for instance, in our case, it comprises Constantan wire, whose free electrode ends $F_1$ and $F_2$ connect up with (through proper resistance) a precision milli-ammeter and a supply of current for heating such as a storage battery, etc.; g is a supply-container to hold the oil with an indicator for filling to a definite level or capacity; this oil chamber surrounds the adsorption vessel; it is circular in shape, and toward the bottom it narrows down to a small diameter, forming the film container h; at the extreme lower portion of h is attached a capillary connecting tube i, which leads to the adsorption space or container.

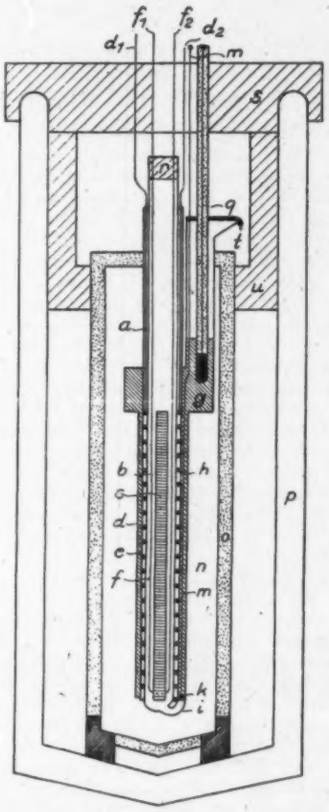


FIGURE 1

Inserted in the connection i, there is fused a small valve or capillary opening k, which may be opened and operated by means of rod m, which allows the oil to flow into the adsorption cavity. The quantity of oil in the film chamber h comprises approximately the oil required for the adsorption space. The quantity of oil in g is about identical to that needed in space h. The supply chamber g, containing the oil could, of course, be so arranged as to actually be a narrow oil chamber surrounding the adsorption cavity, thus doing away with the film-chamber we have been using; in that case the capillary valve can be situated in the oil chamber at the top.

The space n is an evacuated space, surrounding the adsorption space and the oil supply vessel; o is a thermal insulator surrounding vacuum chamber n, which may be made of cork, felt, etc; p is a Dewar flask, q an ordinary thermometer, r, s, t insulating covers and u is a non-conducting isolating-ring. The Dewar vessel itselt is further covered with an insulating layer of cork, felt, etc. It is separated by an air-space when it is finally fitted into a water chamber, which is also covered and insulated with a covering of felt. The

whole arrangement is placed in a cork chamber setup in a room or laboratory kept at constant temperature.

### Procedure

First, a comparison or standardization of the resistance thermometer is made; this is done by charging the apparatus next with ice, then with 20°, 30°, 40°, 60° and 80°C. water, and in the last test, using boiling water; in each case the resistance is measured and noted. We do not calibrate the resistance thermometer, as did Bachman and Brieger in a separate vessel but actually in the apparatus itself. We also did not immerse the wire in the oil.

When this has been done the oil supply or receiver is filled up to the marker (for quantity control) with the oil to be tested, such as a lubricating oil, and a period of time is allowed to pass in order to bring the containers and oil to equilibrium. A blank run is now made to determine the heat-of-wetting of the glass walls and the heating-unit.

One can now make a run with metal, removing previous oil and claining out the oil receiver. The run with metal pieces is made using them in the form of small round rods of 3 mm. diameter and 100 mm. long. One actually need not entirely empty the oil from the oil-supply vessel remaining after the blank run, but may merely suck-back the oil into the receiver-vessel, taking care that the test rod does not touch the oil-covered glass sides or walls when it is inserted into the adsorption cavity. If this is done one need not, in making the actual run, take into account the heat-of-wetting of the glass walls.

After the metal rod has been inserted into the adsorption space or cavity the apparatus is allowed to stand several hours to allow temperature to come to a constant value, and until the variations have been equalized and a steady state has been reached. Readings are now made on the resistance thermometer every 5 minutes or less for about 30 minutes, which is the preliminary period, and records the actual steady state and which values comprise the series of initial readings. Valve K is now opened by means of rod m and the test oil allowed to enter the adsorption space. Temperature readings are made in 3-, 4- or 5-minute intervals, and recorded until they have become steady and absolutely constant. A heat-input of definite amount is now added to the adsorption space by means of the heating unit for a definite time period, and the temperature change and rise thus resulting is again recorded. The electrical energy for supplying heat-input is preferably chosen so that it lies in about the same temperature range as the adsorption heat-rise effect. The calorimeter is thus calibrated. With this setup temperature differences of the order of 1/1000 to 1/10000 degrees Centigrade can be conveniently measured.

When testing metallic powders it is best to proceed as follows: weigh out a definite amount of powder equal to a definite desired volume, and fill the adsorption space in every case to the same and equal height. By doing this one can be certain that the conditions as far as quantity is concerned are the same in each case. This is very important. The presence of the powder in the narrow adsorption cavity, with a definite height, and the entry of the oil from below into the cavity insures the fact that the metal is thoroughly and uniformly soaked with oil; otherwise one may find the powder floating on the surface of the oil, as occurs when the powder is poured from the top into an oil container. The small narrow test-chamber affords the advantage of doing away with stirrers since the heat evolved from the adsorbing surface of the powder travels only a minute distance to the resistance-thermometer. The use of stirrers is condemned as they certainly complicate matters, giving added temperature rises which well obscure the very small increments of heat evolved when we deal with adsorption phenomena.

### Adsorption of Gases

Adsorption in gases or by solution follows in about the same general direction and degree regardless of the influence of the molecular magnitude and the disturbing effects of degree in "ultraporosity" (compare Footnote 4). In the case of adsorption from solution the main consideration is solely that the process of adsorption of an adsorbate is almost exclusively dependent on the solvent. One could possibly add that the greater the adsorption-constant of the solvent in relation to the adsorption-constant of the solute (or adsorbate), other conditions being equal (and when no disturbing factors enter the problem otherwise), the greater will adsorption be retarded in most every case which we have observed. Solubility characteristics and their relationships at various temperatures naturally will modify the acceleration or retardation of all adsorption phenomena. In general, the adsorption of a dissolved substance will become less as the boiling-point of the solvent increases. L. Wohler and W. Wenzel, in a contribution entitled "Report on the Meaning of the Adsorption Equation of Boedeker" (Koll. Zeit. 53, 273 (1930) had established that the adsorption of paraffin from benzines diminished as the boiling-point of their benzines was increased. Benzines of boiling-point 147° C. and 103° C. and a pentane boiling at 38° C. were used.

Ethyl ether, which was also used, acts similarly to a saturated hydrocarbon of the same boiling point in the experiments on the adsorption of paraffin from ether solutions.

### Oils are Mixers

Lubricating-oils must not be considered definite individual hydrocarbons; they comprise a mixture, or more truly, a solution of a whole series of various organic chemical substances, mostly hydrocarbons. The number of these substances is known to be quite large which enter into lubricant oils of petroleum origin. In this connection the following questions will be asked:

(1) To how great a degree are these various components or lubricating oils actually adsorbed? How do the higher-boiling-point components behave in relation to results obtained with the low-boiling-point mixtures? How does chemical constitution of the individual group or the hydrocarbon itself affect adsorption? Do certain molecular structures play an important role in this phenomenon?

(2) What is the relation of the substances in (1) to their function as ture lubricants or oiliness agents?

(3) What practical applications are there for the oils based on conclusions from (1) and (2)?

Let us next discuss the manufacture of lubricants from petroleum and the type of hydrocarbons involved. We note, first, that crude petroleum is a material of exceedingly variable and heterogeneous make-up or composition. For instance, there are those oils where the saturated hydrocarbons, which so characteristically possess long carbon chains, are strongly predominant, and again there are crude oils which contain considerable hydrocarbons of saturated and unsaturated ring structure.

According to H. Herbst active carbon is said to exist in the form of a six-carbon ring[4]; it would therefore appear that that ring and unsaturated compounds should show good adsorption tendencies.

Crude petroleums, furthermore, contain also extraneous compounds, and asphaltic substances, which are removed from the oils by refining processes such as propane precipitation and by sulphuric-acid treating. The author and others working with lubricants have observed that some of the asphaltic substances in petroleum separate out in the form of small dark brown crystalline matter, when the crude is treated with air (air-blowing). These substances, as well as easily resinified materials, must not be present in oils to be sold and used as lubricants, since these substances give rise to and are the cause of sludge-formation in the motor or on the bearing to be lubricated. These deposits finally cause carbonization. Lubricants are normally obtained from crude oils by first driving off by distillation the low-boiling benzine cut of 150°C. boiling-point, followed by the removal of a 150-300°C. cut from which are manufactured various light oils, etc. The residue above 300°C. is the source of the true lubricants. The author carried out investigations (Petroleum 22, 424 (1926), in the adsorption of asphalt from crude oils or its residues by the use of clays containing a considerable amount of hydro-silicates. It was observed that asphaltic material of highest specific gravity was adsorbed first of all, and the oil treated and bleached by the clay showed a lower specific gravity than the original oil. In the case of adsorbable substances it is usually always a question of unsaturation-substances which easily oxidize by means of air, oxygen, etc. By clay-treating and bleaching mineral oils, however, there are also removed some excellent oiliness hydrocarbons and these are lost in the process. For example, an engine-oil distillate shows a higher heat-of-adsorption than does the refined raffinate. The lubricity of oils diminishes considerably by propane and acid

refining. The whole system of refining technique may have to undergo some fundamental changes in order to refine oils and yet preserve their original lubricity or the oiliness which was inherently present before refining.

From my observations I would say the substances of high specific gravity and therefore those containing the larger molecules are adsorbed most tenaciously. It was also observed that in the case of the paraffin and naphthene series the adsorption tendency increases with increasing boiling-point and molecular weight, while the coefficient of friction decreases. There are, of course, organic compounds much more strongly adsorbed than any paraffin.

Substances under discussion must, howerer, be subjected to some sort of systematic evaluation so that new and modern standards may be set up for the whole procedure of refining and also lubrication techniques. The question will arise whether oils which happen to consist of a wide variety of conglomerates of many hydrocarbon series should be used and classified as lubricants or whether true lubricants should be oils consisting only of definitely classified high-molecular-weight substances alone or in mixture with one or with a few definite low-boiling hydrocarbons. One will also find that definite mixtures of various groups will give good results. The adsorption, which is shown by a high-molecular-weight substance toward an adsorbent will be, as we have shown, in the main retarded to a degree directly proportional to the adsorption-constant of the solvent; the greater the latter the more will adsorption be hindered. One would also need to investigate how lubricating efficiency varies with oils which are themselves of high molecular weight and are therefore adsorbable as such in comparison with mixtures of these plus various low-molecular-weight oils, say in various proportions. We see, therefore, that these statements may all lead to various procedures of oil evaluation which I will not discuss singly at this time. Fundamentally, it is important that the metallic field be such that it can, in the case of oil mixtures, actually exert a truly selective action of definite degree upon the individual conponents.

Another phase of our studies would be the problem of relations existing when one adds a solid in a finely divided form or a colloid, such as graphite, to a lubricant and to study the adsorption tendencies of this solid and the oil together and separately. In this work one would first select many types of colloidal substances and determine their adsorption on metal and their lubricating value, and again the adsorption of various oils onto the colloid; and again the adsorption of the colloids and the oils simultaneously on various metals.

One can expect to note from such investigations that the adsorption of colloids on metals is just as pronounced as that of crystalloids on the same surface; this is of great importance in the field of lubrication.

The adsorption of oils on metals[6] is relatively mild compared to that of graphite and similar active carbons.

When working with straight lubricants we must seek and employ oils as well as, when necessary, organic substances which posses high adsorption constants for the particular metal. These adsorption constants of oils for any metal differ quite widely. Colloidal graphite is well adsorbed by metals; since the former adsorbs oils better than do the metals the differences between various oils become less manifest and are more or less annulled.

I would point out that the adsorption of colloids on the surfaces of crystals reaches saturation or equilibrium already with fairly low concentration of the colloid; for this reason one need only use low concentrations and small amounts of these substances. The colloid itself will then also adsorb crystalloidal material so that it takes on, as it were, a two-fold function.

The investigation and discussion of our work on the adsorption ability of metals for various adsorbable substances such as oils, organic derivatives, crystalloids, etc., alone or in the presence of colloids such as graphite, etc., will eventually give rise to new procedures and manufacturing methods. This would be to the end that technologists would present a clearer picture of the technique of lubrication itself, to perfect the present type of petroleum lubricants still further, and finally to develop and produce new definite organic synthetics, as additives for enhancing certain lubricating-oil functions and properties.

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# An Improved Mixing Device For Large Charge Stock Tanks


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MIXING petroleum products in large tanks has generally been done by circulating the contents with existing transfer pumps. Various arrangements of inlet and suction lines, use of distributing spiders or jets in the tank have given more or less satisfactory results. Even the best of such arrangements tie up tank and pump for hours and rarely give a completely uniform mixture. Installation of power-driven mixers often is not unjustified on large process charge stock tanks, or even on finished-product blending tanks.

Some years ago the Paulsboro refinery of Socony-Vacuum Oil Company was faced with the problem of providing uniform quality of feed stock to crude stills running on a blend of three crudes, similar in type but varying widely in viscosity and percent content of the various lubricating-oil cuts. The necessity for a prompt solution of this problem led to the development of a very effective and simple tank mixing device.

The weakness in previous eductor mixers appeared to be that there was no positive means of assuring that all of the tank contents was recirculated through the eductor and remixed with the other portions of the tank liquid and the jet liquid. This weakness was effectively overcome by increasing the ratio of educted quantity to jet quantity over that provided by standard eductor design and by connecting the eductor suction to a standpipe extending to the top of the tank, with ports cut in the side of the standpipe every few inches and increasing in area from the bottom to the top. The eductor was placed horizontally near the tank bottom so that

the discharge struck the shell at an acute angle causing a rotation of the entire body of oil in the tank.

This arrangement was so successful that by filling the tank through the eductor and pumping in the largest volume component of the blend last, the mixture was always so uniform at the end of filling that no additional circulation was required. The other charge tanks of that group were at once similarly equipped and have been in continuous use ever since without any modification, the mixing efficiency being quite satisfactory for the duty involved. These tanks were each 8000-barrel capacity.

Various modifications and different arrangements have been used in later installations to suit particular service conditions, but no change has been required in the fundamental arrangement described above. The suction mixing tube is usually installed near the center of the tank, with the eductor discharging horizontally near the shell.

In studying means of assuring complete recirculation it was found that commercial eductors are usually designed to handle educted quantities about equal to the jet quantities and to provide a considerable head. However, submerged eductors used as tank mixers do not need to produce more head than the friction loss through the system. Keeping this fact in mind, it was found practical to design the eductors with much less lifting power than is usually provided, but with the ratio of educted amount to the jet amount very greatly increased. This increase has been such that when using a circulating pump of reasonable size compared to the tank volume, only a short period of recirculation is required to draw back through the suction mixing tube a volume equal to the entire contents of the tank.

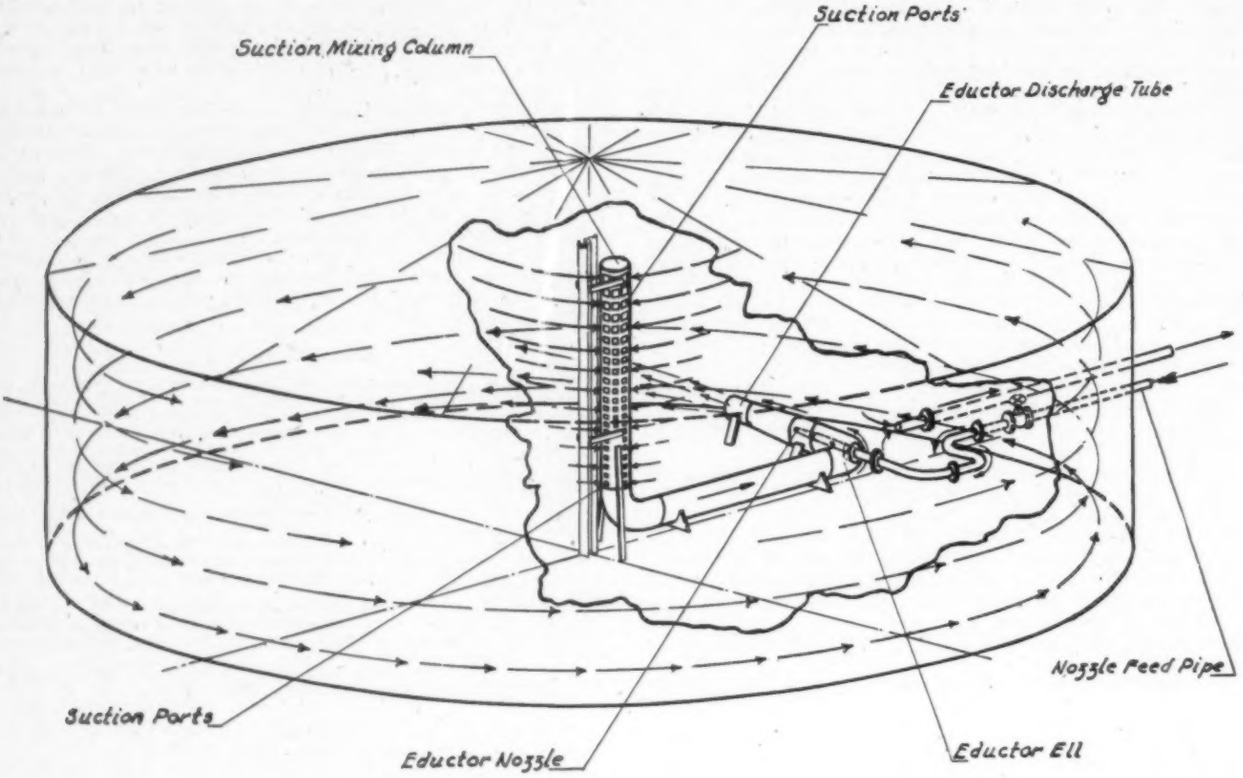
This large educted quantity when drawn from all layers in the tank at once, through the vertical suction mixing tube, and drawing progressively larger volumes from each succeeding higher layer, while at the same time the entire contents of the tank is being rotated, does, in fact, force every part of the liquid to flow back through the mixer and assures absolute uniformity of the mixture in a relatively short mixing period.

In practice it has been found satisfactory to design mixers for still charge, or similar work where absolutely complete blends are not required, with a ratio of educted to jet quantity of 5 or 6 to 1. On these tanks no recirculation is necessary, all mixing being done by filling the tank through the jet. These jets do a satisfactory job, with a drop of pressure of 10 pounds which is usually well within the capacity of existing pumps used for filling the tank.

In preparing finished product blends in large tanks, such as are often used for motor and aviation gasolines, it usually pays to design the eductor and suction mixing tube for much larger ratios, such as 10 or 12 to 1. Thus, with a 10 to 1 ratio in an 80,000-barrel tank, if the filling and circulating pump handles 5,000 barrels per hour, it will draw 50,000 barrels per hour through the suction mixing tube. This quantity will be drawn from some 40 different levels in the tank at the same time, all 40 streams blending together in the suction tube and mixing thoroughly with the jet discharge. Thus, in 1 hour and 36 minutes' recirculation after complete filling, every bit of product in the tank will have been positively blended with a composite of all the rest of the tank.

In such an installation, if the last half of the tank is filled through the jet, the lead and dye added during the same period, and recirculation continued at once while the whole contents is revolving in the tank from the effect of filling through the jet, a completely uniform blend will be assured and the tank will be ready to use in less than two hours after filling.

These mixers are adapted to a wide variety of fluid blending in almost any size tank. They are in satisfactory use in making various chemical blends, as well as on petroleum products from gasoline to fairly viscous lubricating oils.




EDUCTOR TANK MIXER

*The system described herein is covered by U. S. Letters Patent assigned to Socony-Vacuum Oil Company, Incorporated, under which licenses are available.

# A Mercury-Balance Pressure Regulator*

A. J. BAILEY, University of Washington

OF THE numerous pressure-regulator designs reported, the large majority involve elaborate and costly apparatus which permits neither rapid assembly and easy adjustment nor portability and application to different operations involving reduced pressure.

Broadly, pressure regulators can be classified as intermittent or continuous in operation. Most of the elaborate electrical assemblies maintain the pressure at an average value by intermittent pumping, or intermittent leaking with constant pumping. Basically, all these intermittent devices would appear to produce pressures which oscillate above and below the average value, the degree of oscillation depending upon the ballasting effect of the entire system and the sensitivity of the compensating device. Continuous regulators, on the other hand, adjust the amount of leakage so that the sum of gas from the vacuum system plus the controlled leakage equals the capacity of the pump. The precision of pressure regulation then depends on the precision of the proportioning valve—i.e., the sensitivity of the regulator—which is a compound factor determined by the smallest change which causes compensation, the rapidity of compensation, and the magnification of the compensation. Ideally, a continuous regulator should react swiftly and to an exaggerated degree to a minute departure from established pressure.

The designs involving direct control of leakage by mercury balance appeared to offer a precise method of obtaining continuous regulation with all three of these characteristics. The precise types devised by Schierholtz[4] and Thelin[6] used the translation of mercury in a manometer to actuate a lever which controlled the magnitude of the leak. The mercury control of Emerson[3] employed mercury entrained in the stream of leakage to obtain the final degree of throttling. The designs of Swayze[5] and Watts, Riddick, and Shea[7] are examples of intermittent operation of pump and leak, respectively. The apparatus of Caldwell and Barham[2] employed a glass valve operated directly by the mercury in a manometer. Numerous other devices, including many of considerable complexity, have been reported.

## Apparatus

Apparatus patterned originally after those of Schierholtz and Thelin was improved through four successive models. The use of a mercury column to balance the excess pumpage by leakage was found to be precise, rapidly responsive to disturbance of equilibrium, and capable of indefinite magnification of compensation.

Two modifications of the final device are presented here. They employ the same principle as those of Schierholtz and Thelin, but have the mechanical beam eliminated and incorporated in the manometer; one has a loop type of manometer which permits wide range and easy adjustment. Both require only elementary glassblowing technique to construct. The construction is shown in Figures 1 and 2.

Three sizes of tubing were used: 1-mm. capillary, 8 mm. in outside diameter, and 19 mm. in outside diameter, as indicated by **A, B,** and **C,** respectively, in Figures 1 and 2 (lettered identically). The glass manometer was supported in two places: by the spring, **D,** and the pivot, **E.** The spring contained 90 turns of 26-gage piano wire coiled on a 4-mm. rod and was connected by a swivel, **F,** to the thumbscrew, **G,** which adjusted the tension in the spring. Pivot **E** was a glass tube at right angles to the manometer, which served both as the vacuum connection and the shaft about which the manometer rotated. The thumbscrew and pivot were supported by the cold-rolled iron rod, **H,** which was attached to a ring stand by a right-angle clamp. To this rod was attached a rubber stopper, **I,** which served as the valve seat. The stopper was preboiled in dilute alkali and the glass seat was ground flat with a fine pocket whetstone.

## Operation

The regulator in Figure 1 was operated by connecting to the vacuum system with the spring relaxed. When evacuated to the proper degree, the spring was tightened until the

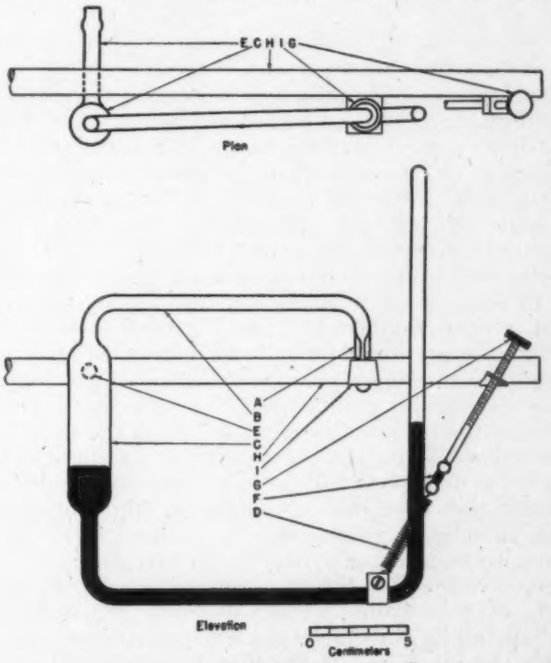



**FIGURE 1**

**Pressure Regulator with Closed Mercury Manometer**

valve barely opened. While the actual opening was not usually easily visible, the effect was noted on a separate manometer. Once adjusted, a decrease in pressure in the system caused the mercury in the manometer to flow into the reservoir, unweighting the manometer-beam and opening the valve. An increase in pressure caused opposite effects.

The regulator in Figure 2 was similar in operation, except that the stopcock was left in the open position while the proper degree of vacuum was established roughly by adjusting the spring tension. The stopcock was closed and the final adjustment made by adjusting the spring tension. A de-

* This article has been printed in Industrial and Engineering Chemistry, **15**, 4, p. 283.

crease in pressure then caused the mercury to flow out of the closed end of the manometer, unweighting the manometer-beam and allowing the spring tension to open the valve.

### Precision of Regulation

The precision obtained with the regulator shown in Figure 1 was about 0.1 mm. of mercury; that of the type in Figure 2 was not readable with the naked eye. Many factors influence precision. The precision obtained with these regulators was the algebraic sum of these variables under given conditions and was by

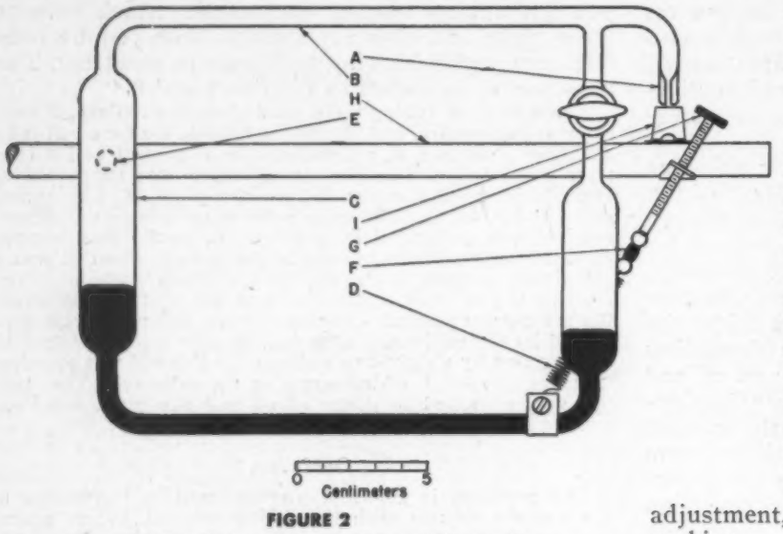



**FIGURE 2**

**Pressure Regulator with Loop Type of Manometer**

no means the ultimate precision of which the device was capable.

Spring tension affected sensitivity greatly. A spring having a small load-deflection ratio was more sensitive than a stiffer spring. The diameter of the tubing in the closed limb of the manometer controlled the weight of mercury shifted for a given pressure change, hence larger tubing in the closed limb of the manometer greatly increased sensitivity; because of location the size of the open limb was unimportant. A bubble of air was always trapped in the closed end of the manometer in the Figure 2 type of regulator; a larger bubble resulted in increased sensitivity. A bubble trapped above the mercury in the Figure 1 type of regulator increased the sensitivity and increased the pressure range over which the device was operative. While the pivot bearing on these regulators was the glass tube, better practice would be to sheath this tube with a machined, split metal bushing, since glass tubing is rarely round and is apt to stick in the bearing. It would likewise be better practice to connect to the vacuum connection, **E**, with a loop of thin-walled rubber tubing, prevented from collapsing by Raschig rings inserted at intervals, and with the other end of the loop fastened permanently to the support bar, **H**, so that torque on the regulator by changing the position of the suction tubing would not change the adjustment of the regulator.

Both regulators were independent of variations in atmospheric pressure. Adjustment to a predetermined pressure by means of the thumbscrew, **G**, having 32 threads per inch, was easily effected. Both regulators controlled pressure with uniform facility over the entire pressure range. Both regulators were susceptible to "bouncing" under certain conditions—i.e., an oscillation of the mercury column and concomitant movement of the manometer-beam, causing intermittent admission of air. It was found to be due to parallelism of valve seat and face, and was eliminated by an imperceptible bending of the valve seat support, so that valve seat and face were not quite parallel and hence the valve could not close completely. The bouncing apparently was due to complete closure of the valve. In order to open the valve, pushing against atmospheric pressure required overcompensation of the mercury-balance, and then rapid overcompensation in the opposite direction resulted owing to the sensitivity of the device, again causing sealing of the valve. This bouncing phenomenon has been noted in other regulating devices[1] using a mercury column and is due to similar causes.

Generally speaking, the Figure 2 type of regulator was more flexible and sensitive, but no stopcock under vacuum can be considered completely dependable under long periods of operation. Either regulator may be made capable of increased sensitivity by increasing the diameter of the tubing in the closed manometer limb, lengthening the beam, using a more elastic spring, or trapping a larger volume of air in the closed end of the manometer. Where great sensitivity is not required, a rubber cushion can be used to replace the spring suspension, but all attempts to use rubber have necessitated continual adjustment, owing to the fatigue of the rubber cushion.

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[3] Emerson, R. L., and Woodward, R. B., *Ibid.*, **9**, 347 (1937).
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**DECIMAL EQUIVALENTS OF COMMON FRACTIONS**

| 32nds | 64ths | Decimal Fraction | 32nds | 64ths | Decimal Fraction |
|---|---|---|---|---|---|
| | 1 | .015625 | | 33 | .515625 |
| 1 | 2 | .03125 | 17 | 34 | .53125 |
| | 3 | .046875 | | 35 | .546875 |
| 2 | 4 | .0625 | 18 | 36 | .5625 |
| | 5 | .078125 | | 37 | .578125 |
| 3 | 6 | .09375 | 19 | 38 | .59375 |
| | 7 | .109375 | | 39 | .609375 |
| 4 | 8 | .125 | 20 | 40 | .625 |
| | 9 | .140625 | | 41 | .640625 |
| 5 | 10 | .15625 | 21 | 42 | .65625 |
| | 11 | .171875 | | 43 | .671875 |
| 6 | 12 | .1875 | 22 | 44 | .6875 |
| | 13 | .203125 | | 45 | .703125 |
| 7 | 14 | .21875 | 23 | 46 | .71875 |
| | 15 | .234375 | | 47 | .734375 |
| 8 | 16 | .25 | 24 | 48 | .75 |
| | 17 | .265625 | | 49 | .765625 |
| 9 | 18 | .28125 | 25 | 50 | .78125 |
| | 19 | .296875 | | 51 | .796875 |
| 10 | 20 | .3125 | 26 | 52 | .8125 |
| | 21 | .328125 | | 53 | .828125 |
| 11 | 22 | .34375 | 27 | 54 | .84375 |
| | 23 | .359375 | | 55 | .859375 |
| 12 | 24 | .375 | 28 | 56 | .875 |
| | 25 | .390625 | | 57 | .890625 |
| 13 | 26 | .40625 | 29 | 58 | .90625 |
| | 27 | .421875 | | 59 | .921875 |
| 14 | 28 | .4375 | 30 | 60 | .9375 |
| | 29 | .453125 | | 61 | .953125 |
| 15 | 30 | .46875 | 31 | 62 | .96875 |
| | 31 | .484375 | | 63 | .984375 |
| 16 | 32 | .5 | 32 | 64 | 1. |

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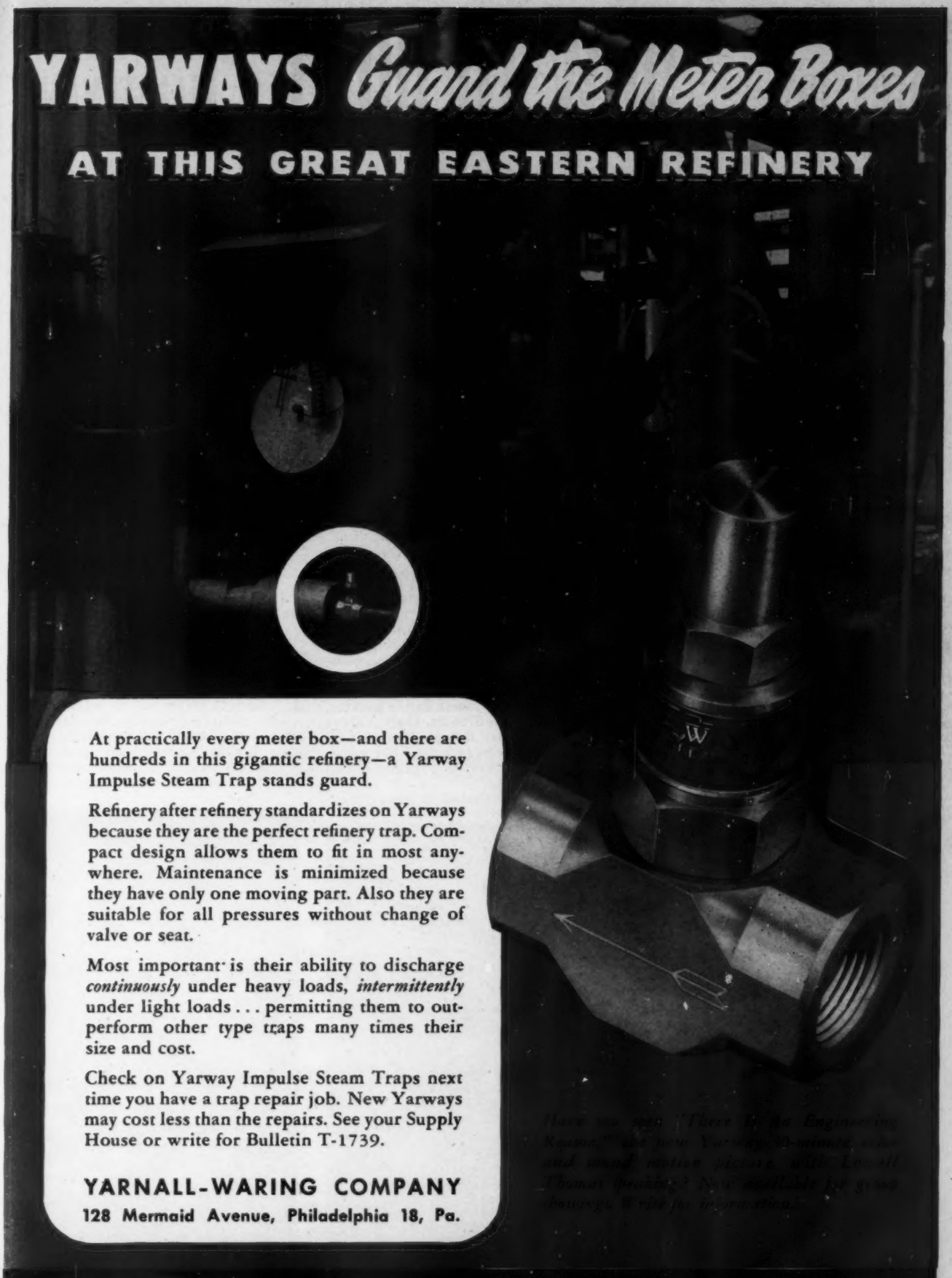

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# OPERATION HOW TO DO IT MAINTENANCE

## 1. Two-Stage Compressor Converted to One-Stage Without Plant Shutdown

THIS problem arose when additional drilling and increased oil flow in the field supplying the gasoline plant so increased the quantity of gas to be processed that more compressor capacity was required. The plant was designed to salvage tank and accumulator vapors by two-stage compression, which is the orthodox method of handling such gases. But by changing the operation of the gasoline accumulator tanks and vapor receivers in the plant to carry about the the same pressure as the distillation equipment, the pressure on these vessels was raised to a point that single-stage compression might be followed with adequate condensation of the desirable gases.

A further problem arose in the desirability of converting from two-stage to single-stage compression without shutdown of the plant.

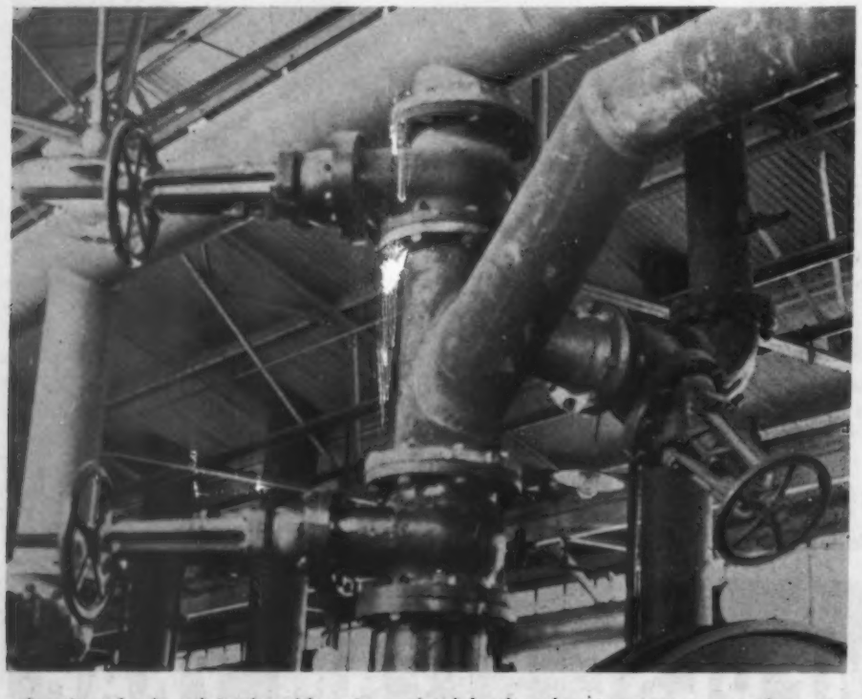
Ordinary practice which might be followed when converting to single-stage with the released low-pressure cylinder attached to the main suction header would have been to shut down the engine room and make the required pipe connections. But a shut-down would have resulted in lowered oil production and a great loss of gas and the condensate manufactured from it. Steps were taken to make the conversion while operating.

The main header runs the entire length of the engine room, above the compressors. One of the neighboring low-stage compressors operating on field gas was fitted on the riser with a block valve under the main header. This cylinder was run on the by-pass manifold, while a spool was made up containing an angle welded connection, reaching across the cylinders between, to the cylinder which was being converted to field service.

Measurements were made carefully so that the fabricated connecting pipe would drop into place, matching companion flanges and all pipe outlets, so that all that was required to make the connections was to insert flange gaskets, insert bolts and tighten up. Only a short time was necessary to make the connections, as the pipe was built from measurements made before shutting down any of the engines.

The result is reflected in the field capacity being increased by the volumetric capacity of the low-pressure cylinder, and substantially the same quantity of recompressor production as had been produced by two-staging the vapors from the accumulators and gas receivers.

—H.A.H.



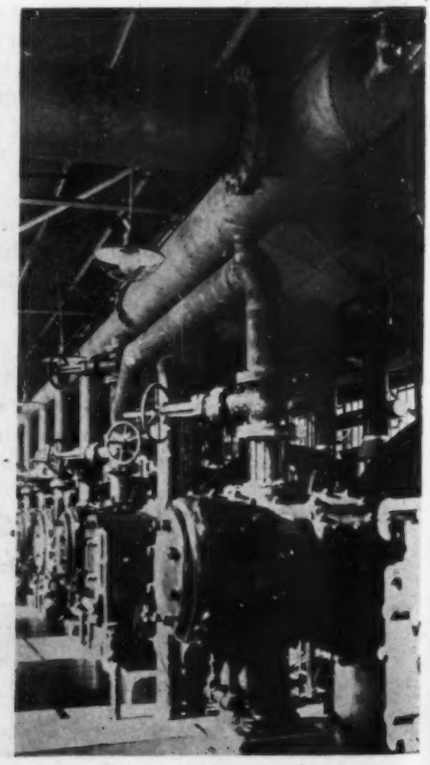
Junction of released intake with main overhead header when two-stage compression was changed to single stage. The view at right shows the cross-over piping.

**THE WINNER**

**A number of readers selected at random and asked to evaluate items printed in this department in December have indicated the award should go to Item 7—Angle Iron Extensions Aid in Pipe Welding. Accordingly, a $25 Victory Bond has been sent to the contributor.**

## 2. Ingenuity Provides Emergency Relief Valve

FABRICATION of a valve which would withstand service pressures and surges, yet which would unfailingly relieve overload on the water system was a problem met in a unique manner by one plant when it proved impossible to



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Emergency Relief Valve

locate a spring-loaded valve in any available stocks. A standard rising-stem globe valve of the required strength to meet pressure conditions was taken and the threads bored out of the bonnet so that the stem would slide freely within the area formerly occupied by threads. The stuffing box, gland and packing of the gland served as guide in the new set-up. The shortened stem was brazed to a transverse section of bar steel, this latter having 1-1/16-inch holes bored through it near the ends.

When the valve had been mounted on the outlet, a nipple was screwed tightly into the discharge end—the valve being placed so that the pressure was *under* the disc—and cylindrical guides welded to line and nipple at the spacing provided by the holes through the Tee-head on the valve stem.

A second bar, placed above heavy springs, served as means for adjusting the pressure on the disc, adjustment being made through threading the outer ends of the guide rods and using spacers to take up part of the "squeeze" in the springs.—B.E.S.

# 3. Calibrating Horizontal Cylindrical Tanks With Ellipsoidal Heads

THE FOLLOWING outline of a method of calibrating horizontal cylindrical tanks was the result of the author's knowing of a laborious and time-consuming calibration of a group of tanks by filling them with water and measuring it out. Upon attempting to find the formula for volume of ellipsoidal heads in terms of the liquid height in various handbooks, it was decided that this particular formula had not been published, so a formula for this volume was developed and a convenient outline of the calculation set up. With the aid of this outline and a calculating machine, one may calibrate a given tank in a few hours.

It is suggested that the "h/d" values are set up from the attached tables, the number of points being determined by the degree of precision required in the calibration curve. The values in the tables are spread out in such a manner as to give more points in the region where the curve is changing slope rapidly, and fewer points where the curve is practically straight. There are 160 values listed in the tables, so if 80 points are desired, use every other h/d value; if 40 points are desired, use every fourth h/d value, etc.—E.E.McR.

### Tank Calibration for Horizontal Cylindrical Tanks With Ellipsoidal Heads Versus Level

(1) Determine;

$\frac{\pi}{z} = \frac{3.14159}{z}$, where z is the ratio of major to minor axis of the ellipse. ($z = a/b$, a = major axis, b = minor axis)

d = diameter of the tank, and d/2, radius of tank.

$d^2$ = square of diameter of tank.

L = length of cylindrical portion of tank from tangent to tangent, in the same units as d and h.

k = conversion factor from cubic units to liquid volume units.

| | Values of k | |
|---|---|---|
| Units of h and d | 42 gal. barrels | U. S. gallons |
| inches | 0.00010307 | 0.004329 |
| feet | 0.17811 | 7.48052 |

(2) Tabulate as follows:

h/d | $A/d^2$ | A | $V_c$ | h | h/3 | $\left(\frac{d}{2} - \frac{h}{3}\right)$ | $h^2$ | $\pi/zh^2$ | $V_e$ | $V_t$ | $V_{lv}$

where h/d values are set up depending on the number of points on the calibration curve that are desired,

$A/d^2$ values are read from the attached table,

$A = A/d^2 \times d^2$,

$V_c = AL$ = the volume of the cylindrical portion of the tank,

$h = h/d \times d$ = height of liquid level in the tank,

$V_e = \frac{\pi}{z} h^2 \left(\frac{d}{2} - \frac{h}{3}\right)$ = volume in both ellipsoidal heads,

$V_t = V_c + V_e$ = total volume in cubic units, and,

$V_{lv} = kV_t$ = volume in liquid units.

(3) Plot $V_{lv}$ versus h for the tank calibration curve.

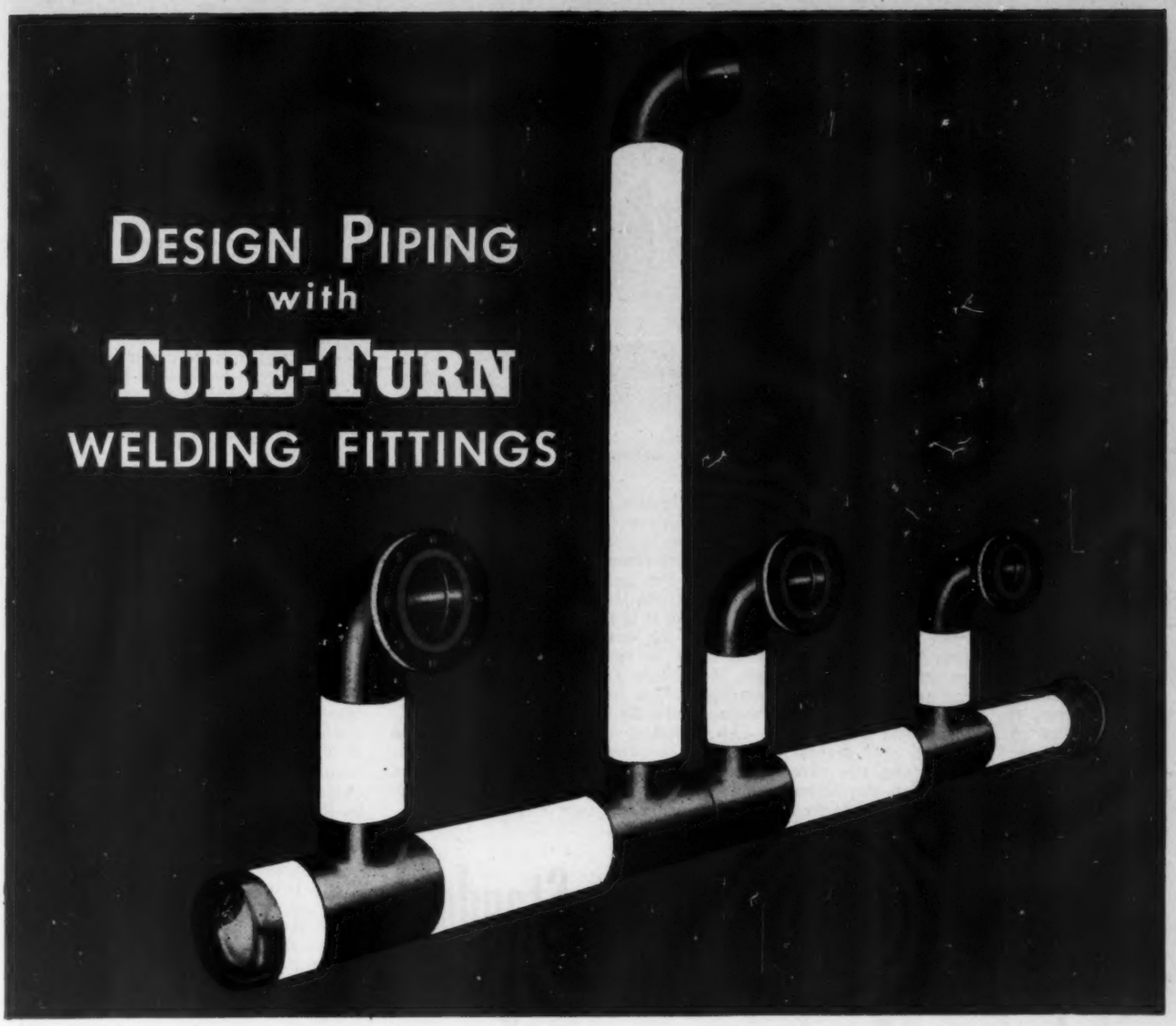
**TANK CALIBRATION—$A/d^2$ Values**

| h/d | $A/d^2$ | h/d | $A/d^2$ | h/d | $A/d^2$ | h/d | $A/d^2$ |
|---|---|---|---|---|---|---|---|
| .001 | .00004 | .210 | .11990 | .620 | .51154 | .915 | .75321 |
| .002 | .00012 | .220 | .12811 | .630 | .52122 | .920 | .75597 |
| .003 | .00022 | .230 | .13646 | .640 | .53085 | .925 | .75864 |
| .004 | .00034 | .240 | .14494 | .650 | .54042 | .930 | .76123 |
| .005 | .00047 | .250 | .15355 | .660 | .54993 | .935 | .76374 |
| .006 | .00062 | .260 | .16226 | .670 | .55937 | .940 | .76616 |
| .007 | .00078 | .270 | .17109 | .680 | .56873 | .945 | .76849 |
| .008 | .00095 | .280 | .18002 | .690 | .57802 | .950 | .77072 |
| .009 | .00113 | .290 | .18905 | .700 | .58723 | .995 | .77285 |
| .010 | .00133 | .300 | .19817 | .710 | .59635 | .960 | .77486 |
| .012 | .00175 | .310 | .20738 | .720 | .60538 | .965 | .77676 |
| .014 | .00220 | .320 | .21667 | .730 | .61431 | .970 | .77853 |
| .016 | .00268 | .330 | .22603 | .740 | .62314 | .972 | .77921 |
| .018 | .00320 | .340 | .23547 | .750 | .63185 | .974 | .77985 |
| .020 | .00375 | .350 | .24498 | .760 | .64046 | .976 | .78048 |
| .025 | .00523 | .360 | .25455 | .770 | .64894 | .978 | .78108 |
| .030 | .00687 | .370 | .26418 | .780 | .65729 | .980 | .78165 |
| .035 | .00864 | .380 | .27386 | .790 | .66550 | .981 | .78193 |
| .040 | .01054 | .390 | .28359 | .800 | .67358 | .982 | .78220 |
| .045 | .01255 | .400 | .29337 | .805 | .67756 | .983 | .78246 |
| .050 | .01468 | .410 | .30319 | .810 | .68150 | .984 | .78272 |
| .055 | .01691 | .420 | .31304 | .815 | .68540 | .985 | .78296 |
| .060 | .01924 | .430 | .32293 | .820 | .68927 | .986 | .78320 |
| .065 | .02166 | .440 | .33284 | .825 | .69309 | .987 | .78343 |
| .070 | .02417 | .450 | .34278 | .830 | .69686 | .988 | .78365 |
| .075 | .02676 | .460 | .35274 | .835 | .70060 | .989 | .78387 |
| .080 | .02943 | .470 | .36272 | .840 | .70429 | .990 | .78407 |
| .085 | .03219 | .480 | .37270 | .845 | .70793 | .991 | .78427 |
| .090 | .03501 | .490 | .38270 | .950 | .71153 | .992 | .78445 |
| .095 | .03791 | .500 | .39270 | .855 | .71507 | .993 | .78462 |
| .100 | .04087 | .510 | .40270 | .860 | .71857 | .994 | .78478 |
| .110 | .04701 | .520 | .41270 | .865 | .72201 | .995 | .78493 |
| .120 | .05338 | .530 | .42268 | .870 | .72540 | .996 | .78506 |
| .130 | .06000 | .540 | .43266 | .875 | .72874 | .997 | .78518 |
| .140 | .06683 | .550 | .44262 | .880 | .73202 | .998 | .78528 |
| .150 | .07387 | .560 | .45256 | .885 | .73524 | .999 | .78536 |
| .160 | .08111 | .570 | .46247 | .890 | .73839 | 1.000 | .78540 |
| .170 | .08854 | .580 | .47236 | .895 | .74149 | .... | ..... |
| .180 | .09613 | .590 | .48221 | .900 | .74453 | .... | ..... |
| .190 | .10390 | .600 | .49203 | .905 | .74749 | .... | ..... |
| .200 | .11182 | .610 | .50181 | .910 | .75039 | .... | ..... |

Linear interpolation is accurate for whole thousandths of h/d values from .100 to .900.

# 4. Concrete Forms Made From Roll Roofing

WHERE several warehouse buildings were to be built about two feet from the ground, one company used roofing material held by wire loops as forms for the concrete piers. By taking a piece of roofing about six feet long and rolling it into a cylinder, and then placing

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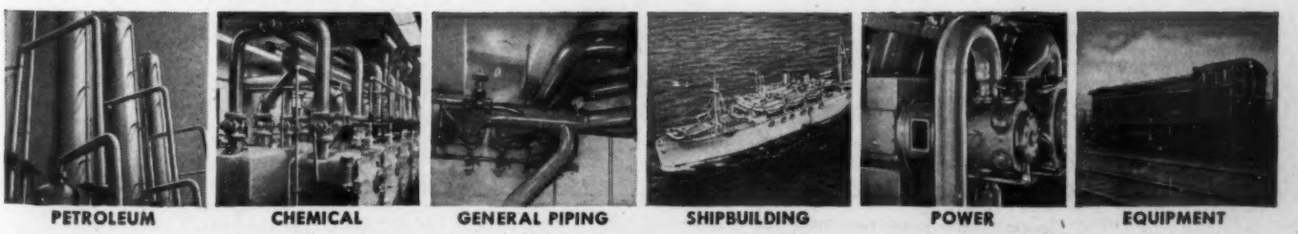

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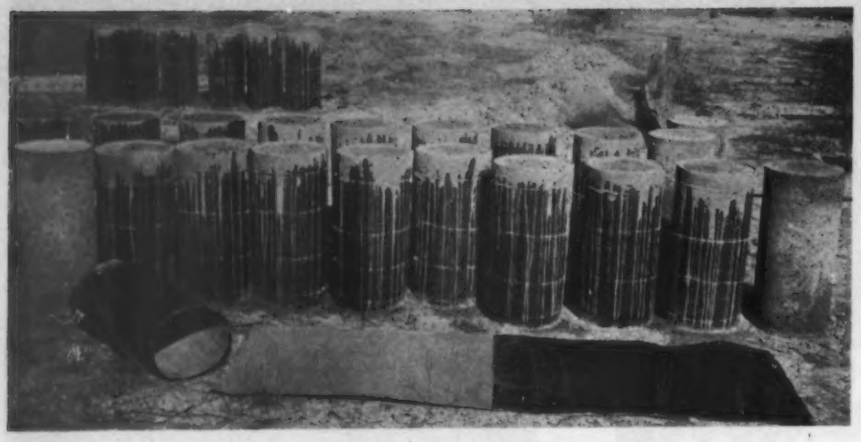


Roofing forms made from asbestos roofing

wire loops around it, a simple form was made for molding the concrete posts. The cylinders were first set on end and neat cement grout poured in the bottom for a depth of approximately two inches. By the time the set of post bases are poured, the first one has attained an initial set so that it can be filled with concrete without further sealing of the bottom. When the concrete has set, the wire loops are removed and the roofing unrolled from the cylinder of concrete.

Each piece of material and the wire loops can be used over and over several times. Between periods of use, the wire loops are hung on a cleat in the warehouse and the roofing material is stacked flat.

By using the same pieces, but rolling them lengthwise, long thin posts may be formed. By using longer strips, posts suitable for fencing may be poured.

When the piers must be cast some distance from the point where they are to be used, it is possible to transport the units at the time of the initial set if the forms be retained in place to serve as protection against impact and the transportation be accomplished without undue vibration.

The roofing material also serves as insulator to prevent chilling of the concrete, and if a cover disk be used, the piers will cure satisfactorily when otherwise freezing damage might occur.

—H. H.

**EACH month the operating personnel in the refining, cycling and natural-gasoline plants determine the winner of a War Bond, maturity value $25.00.**

**Superintendents, assistant superintendents and foremen (subscribers as well as non-subscribers) become a jury to select the most practical contribution to this department on maintenance and operation. The editors rotate voting among men qualified to determine the rating.**

**This award is in addition to payment for the article. The methods of solving plant operation and maintenance problems come from the personnel of the industry. Items provided by Petroleum Refiner staff members will not be eligible.**

**Send contributions to Petroleum Refiner, P. O. Box 2608, Houston 1, Texas.**

# 5. Steam Ejector Made From Standard Pipe Fittings Serves in Emergency

A STEAM ejector may be made from standard pipe fittings as shown in the accompanying sketch. Such an ejector has a capacity of approximately 40 to 50 gallons per minute of water at a suction head of 12 to 15 feet when using 150-pound steam. Lowering the steam pressure will reduce the capacity of the ejector.

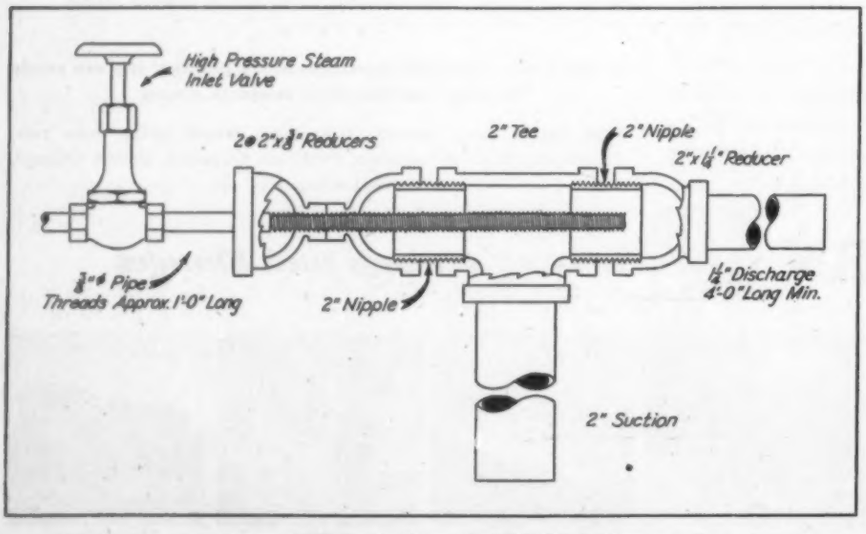
To build the ejector a thread approximately 1 foot long is first made on a piece of 3/8-inch pipe. A reducer of any size is then screwed on to the 3/8-inch pipe followed by a small gasket and a 3/8- x 2-inch reducer. The first reducer is put on backwards so that the 3/8-inch ends of the two reducers are against each other.

The balance of the ejector is then assembled as shown in the sketch.

For best results it is important to have the tip of the 3/8-inch nozzle in the proper position which is usually just inside the 2 x 1½-inch reducer at the right of the sketch. Adjustments in the position of the nozzle tip may be made by loosening the reducer at the extreme left of the sketch which is used as a lock nut to lock the position of the nozzle tip.

The efficiency of the ejector is very low unless the heat supplied to the liquid being pumped is utilized. In this case the efficiency is nearly 100 percent. The ejector has the advantage over the conventional steam pump in being able to pump scale, mud, rocks or anything else that will pass through the discharge pipe.

On one occasion during the war, a sump pump failed that was draining all the water from a large laboratory. Such an ejector was made and used as an emergency sump pump until replacement parts could be obtained for the regular pump. The ejector handled approximately 50 gallons per minute at a suction lift of 12 feet. The ejector operated continuously for about 3 weeks with only occasional attention.—W. R. S.




Steam ejector made from standard pipe fittings

# 6. Reinforcing Drop Legs Minimizes Vibration

WHEN overhead mains are used for collecting and distribution circuits at the plant, it is frequently necessary to bring individual supply lines and drains to ground level for further use. This long drop-leg, being welded in at the base of the overhead run, is sometimes subjected to severe vibrational stress which, in connection with the location of the weld at a point where liquids most readily gather, subjects the joint to a combination of stress and electrolytic corrosion, and unless safeguarded, the life of the joint is found to be much less than that of adjacent piping.

One plant brings the drop-legs to

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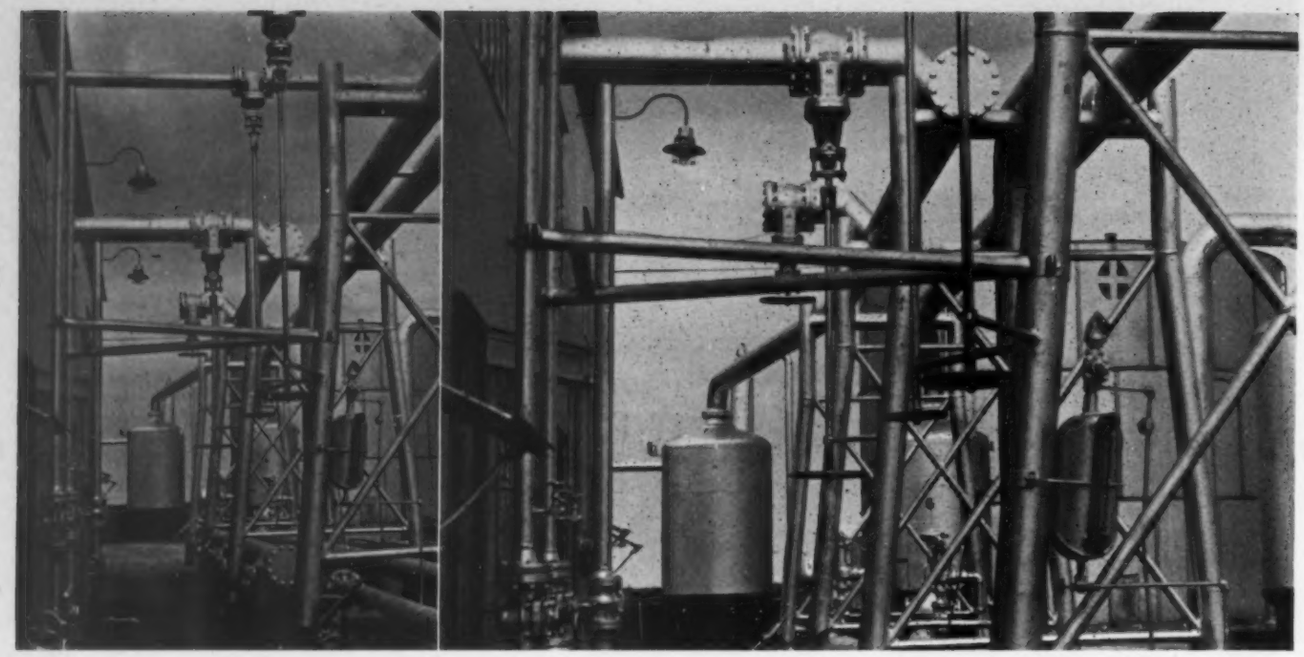


**Two views of the same installation. The larger picture at the right shows more detail of how the reinforcements are installed.**

working level at points where the overhead loop is carried on welded pipe supports, utilizing the upright of the support as anchorage for the smaller vertical pipe in the plant's operating system. Midway between overhead weld and lower support of the drop-leg, a brace is clamped around the pipe, the other end being bolted with a collar to the support, while a third section of pipe, welded between this and an adjacent brace, makes a triangular support which effectively snubs vibration and eliminates undue stress on the welded junction.—E.S.

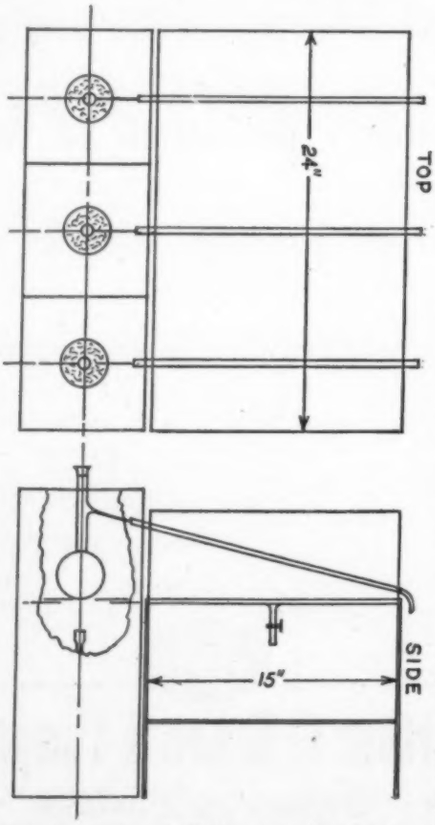
**READERS ARE URGED TO CONTRIBUTE items to this department. Drawings or photographs are desired where practical. Payment for items accepted will be on a basis that will compensate for the time involved and in addition each contribution will be eligible to receive the bond award as outlined in the announcement in page 134. Jury awards will be announced in the second issue after publication.**

# 7. Engler Distillation Units in Batteries

SETTING Engler distillation units in batteries instead of separating them around the laboratory greatly simplifies the supervision of the runs, and also provides the desired uniform condensation conditions with a minimum of handling of cracked ice, and much greater effectiveness in packing the condenser tubes.

Each flask is set in a separate compartment, three being grouped within one housing but with inner baffles to minimize drafts and concentrate the heat where desired. From each of the housings is run a condenser tube, at the angle specified by ASTM rules, three being carried in parallel across a single cooling bath and discharging at a convenient height outside the ice-filled tank. The tank is provided with drain so that the waste water may be drawn off as often as indicated by visual inspection of the bath.

With the distillation units thus grouped in sets of three, the operator can check three as readily as before one could be observed, and it is common practice to run nine distillations in three such groups, with no more supervision or care being required than was the case when three single Engler units were being operated separately.—G.A.T.




**Grouping Engler distillation units in batteries simplifies supervision and insures uniform condensation conditions.**

# 8. Relief Valve Weather Hood

TO protect arm-and-weight-operated relief valves, and prevent water from collecting around the stem and in the valve case, which might freeze and rupture the body casting, one company built weather hoods for valves which might be damaged. The hoods are made of medium-weight galvanized iron, built in the form of a cylinder with one end open. The closed end is made by welding a circular piece of iron to the cylinder, and part of the cylinder seam welded tight. An opening of ample size is made for the valve arm to oprate without striking the hood. So that no moisture will run down the slanting arm when raised from the horizontal, each hood is equipped with an automobile emergency brake rubber weather hood, secured to the cylinder with stove bolts. The valve cover is fastened to the body of the valve by clamping. Two lugs are fastened to opposite sides of the slitted opening, below the rubber cover, which are drilled for a long stove bolt to draw the cove rtightly against the body of the valve.—F.T.H.

# THE MONTH IN THE INDUSTRY

## Western Petroleum Refiners to Hold First Postwar Meeting, Fort Worth March 25-27

Technical sessions, business meetings and entertainment will make up the program for the annual meeting of the Western Petroleum Refiners' Association, Blackstone Hotel, Fort Worth, March 25, 26 and 27. This will be the first convention since the war, although one-day business sessions were held in the war years.

Technical meetings will be held each morning with the general sessions in the afternoon.

Chairman for the first general session will be Roland V. Rodman, vice president of Bay Petroleum Company, Denver. Colonel E. O. Thompson, Texas Railroad Commission, will deliver the address of welcome.

Robert Ash, Washington counsel for Prentice-Hall, Inc., New York, will talk on "Tax Prospects in 1946." Ash is a member of the council of the section on administrative law of the bar association of the District of Columbia. He is the author of "Preparation and Trial of Tax Cases," published by the American Bar Association and Practicing Law Institute. He is well known as an authority on taxes affecting the oil industry, having lectured on several occasions under the auspices of the above organizations in various cities.

R. R. Wason, president of the National Association of Manufacturers, New York, will present the manufacturer's views for the future to the members on Tuesday afternoon. Wason has spent the better part of his business career in the manufacturing branch of the industry. He is president of Manning, Maxwell & Moore, Inc.

On Wednesday, March 28, those attending the general session will hear W. J. Brons speak on "How Good Is Debt." Brons is chief editorial writer of "The Round Table" column, a daily feature in the Chicago Journal of Commerce.

Also on Wednesday afternoon's program will be A. J. Pettigrove, member of a petroleum mission recently sent to Japan and Southwest Pacific to study Japanese oil installations. Pettigrove is a refinery man and during the war years was affiliated with the Associated Refineries at Duncan, Oklahoma. Prior to that time he was with Phillips Petroleum Company and now is connected with Stanolind Oil & Gas Company, Tulsa.

The first technical program will be held Tuesday morning when W. F. Sims, Panhandle Refining Company, will preside. F. Duane Fuqua, Ben Franklin Refining Company, Ardmore, will present a paper, "Crude Oil De-Salting in Refinery Operations." His discussion will present the methods used in combining heat and pressure to remove 92 percent of total salt. The other paper on this program will be "The New U.O.P. M.S. Fluid Catalytic Cracking Unit," by Davis Read of Universal Oil Products Company, Chicago. A small glass working model will be used to show catalyst flow and other operating procedure.

Two technical papers will be presented on the technical program Wednesday morning when M. W. Conn, Phillips Petroleum Company, will preside. John S. Ewing, Carnegie-Illinois Steel Company, Pittsburgh, will discuss "Stainless Steel and Its Application to the Refining Industry." The other paper will be "Simplicity and Flexibility of Thermofor Catalytic Cracking," H. D. Noll and K. G. Holdom, Houdry Process Corporation, and Eric Bergstrom, Socony-Vacuum Oil Company.

Meetings of the diesel-fuel committee and a joint session of the motor-fuel and tractor-fuel committees will be held Monday morning. That afternoon, the finance committee will meet. Directors and officials will be elected Tuesday afternoon.

On Monday evening Ethyl Gasoline Corporation will be hosts at a buffet supper starting at 6:30 p.m. in the ballroom of the Blackstone Hotel. A buffet dinner and Annual Spring Roundup party will be given on Tuesday evening, by the supply and transportation companies serving the refining industry. The annual dinner and dance of the association on Wednesday night will conclude the entertainment. All social functions are planned to include the wives of those attending and it is expected that many will be present.

### Conventions

**March**
- 25-27—Western Petroleum Refiners Association, annual meeting, Blackstone Hotel, Fort Worth.
- 31—American Institute of Chemical Engineers, South Texas Section, Rice Hotel, Houston (through April 3).

**April**
- 17-19—Natural Gasoline Association of America, Baker Hotel, Dallas.
- 18-19—National Petroleum Association, Hotel Cleveland, Cleveland.
- 23-25—Southwest Gas Measurement Short Course, University of Oklahoma, Norman.
- 28—API Directors, Blackstone Hotel, Fort Worth, Texas.

**May**
- 7-9—National Association of Corrosion Engineers, President Hotel, Kansas City.

**June**
- 24-29—American Society for Testing Materials, annual meeting, Buffalo.

**September**
- 16-20—National Instrumentation Conference and Exhibit, Wm. Penn Hotel, Pittsburgh.

**November**
- 11-14—American Petroleum Institute, annual meeting, Stevens Hotel, Chicago.

### Sinclair Building New Laboratory at Chicago

Sinclair Oil Corporation has announced plans for immediate construction of an extensive petroleum research laboratory at Harvey, Illinois. The building and other facilities will provide new and enlarged quarters for the research and development department which now is located at the company's refinery in the East Chicago area. The laboratories are the only part of the company's facilities which will be moved.

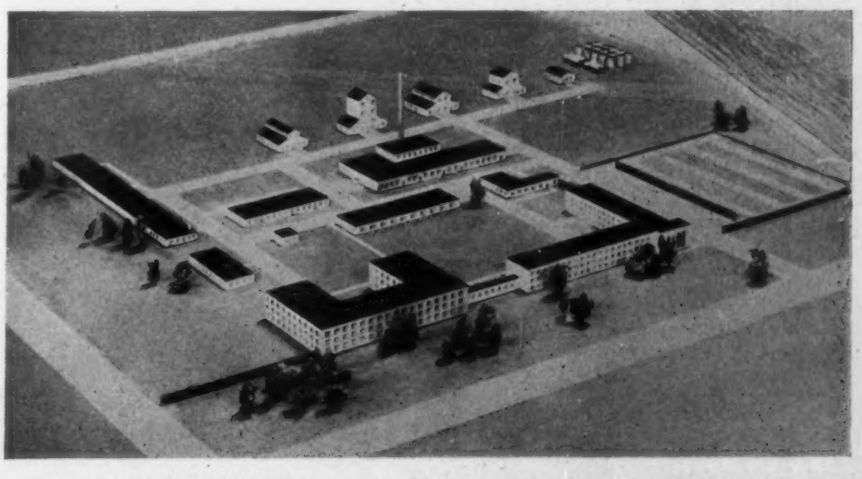
The project at Harvey covers ample acreage and is conveniently located within 20 miles of downtown Chicago. It is near enough to the present location of the laboratories that personnel may retain their present places of residence.

When completed, the size of the laboratory practically will be doubled, and the technical and professional staff will have more than 550 people, it was said.

### Walter Miller Retires as Continental Refinery Head

With reitrement of Dr. Walter Miller, vice president, Harold G. Osborn, manager of manufacturing, has assumed full charge of the eight refining plants of Continental Oil Company. Miller retired March 1. He will continue active in the industry as a consultant in refining.

Other changes in Continental Oil Company's management announced include appointment of E. F. Batterson as assitsant to the president, where he succeeds George D. Olds, resigned, and appointment of F. B. Haverfield as man-



Model of Sinclair Oil Corporation's research and development laboratories at Chicago

# THE MONTH . . .

F. B. HAVERFIELD

ager of the natural gasoline and gas division, to succeed R. A. Carter, resigned.

Dr. Miller has been connected with the industry since completing his elementary schooling in Elizabeth, New Jersey. In 1909 he went to work in the Bayonne plant of Tide Water Oil Company as a yield clerk and continued his studies in night school giving special attention to chemistry and engineering. He had become process superintendent at Bayonne when he decided to move to the Mid-Continent area. He was with Pierce Petroleum Corporation as superintendent of its three refineries for a brief period and joined Cosden and Company, Tulsa. When he became a consultant in 1921 one of his clients was Marland Oil Company, which he joined in 1922 as manager of refining. When the Marland and Continental Oil Companies were merged in 1929 he was elected vice president in charge of manufacturing.

Miller is a member of the American Chemical Society, the American Society of Mechanical Engineers, the American Institute of Mining and Metallurgical Engineers and the American Society for Testing Materials, as well as the British Institution of Petroleum.

Osborn joined Continental Oil Company in 1921 in its power division. A year later he became office assistant to the vice president. In 1926 he was made assistant superintendent of the Ponca City plant, assistant manager of manufacturing in 1926, and manager in 1935.

Haverfield joined the company in 1929 as plant chemist at Big Lake, Texas, and previously had for nine years been with Signal Oil Company in California. He was made assistant superintendent of the gasoline department in 1939 and has been assistant manager since 1943. He will be succeeded as assistant manager by D. B. Edwards, who in nine years with the company has been chemical engineer, process engineer, and assistant to the manager. F. B. Willson has been made assistant to the manager.

Olds has been assistant to the president since he joined the organization in 1933. He will continue for a short time as president of Petroleum Chemicals, Inc., and Oil and Gas Research, Inc., it was said.

Carter will be associated with the manufacturing department of Stanolind Oil & Gas Company, and will have charge of sale and purchase of natural gas and sale of liquid products, with headquarters at Tulsa. He had been with Continental Oil Company 22 years.

## E. W. Luster Heads Esso Engineering Consolidation

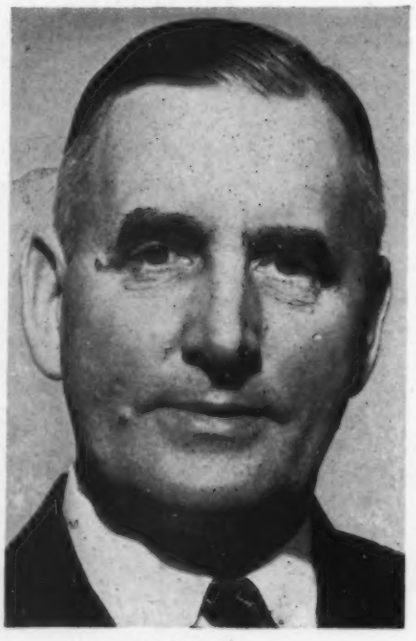
In a move designed to give greater emphasis to mechanical development and engineering research, Standard Oil Development Company, central technical organization of Standard Oil Company (New Jersey), has consolidated its two engineering departments.

The consolidation brings into one organization more than 400 scientists and other workers of the general engineering department and the process engineering department which have operated independently for nearly 10 years. The new organization, named Esso Engineering Department, is directed by E. W. Luster, chief engineer, who reports to Dr. N. E. Loomis, vice president in charge of engineering.

The regrouping, Dr. Loomis said, is expected to place greater attention on mechanical engineering and engineering research work, improve work supervision and increase efficiency. The department's activities also will be expanded to cover not only refining, but certain engineering problems in other lines.

Other objective of the consolidation, Dr. Loomis said, include better coordination of technical work, faster handling of approximate designs and estimates, expansion of the process test division to include testing of mechanical equipment, and greater attention to mechanical engineering economics.

Under the new setup, E. H. Barlow, former chief engineer, becomes consulting engineer of Standard Oil Development Company, handling many of the contacts with company executives on engineering matters of general interest. Assistants to Luster are P. J. Byrne of the process engineering department; C. E. Paules of the general engineering department, and N. F. Myers, former associate manager of the process division.



E. H. BARLOW

E. W. LUSTER

C. T. Harding is named chief process engineer, C. F. Hesselbach, chief mechanical engineer, and A. J. Ely, chief construction engineer. As part of the reorganization, a group of project engineers including G. W. Bauer, E. H. Clendenin, P. J. Harrington, C. A. Hillman, E. H. Kares and J. R. Schonberg, will serve as coordinators on major projects.

## Montgomery Advanced by Standard of Indiana

S. A. Montgomery, manager of the Whiting plant of Standard Oil Company (Indiana), has been promoted to assistant general manager of manufacturing in the Chicago general office. He was succeeded by Dr. W. J. McGill, manager of the company's Wood River refinery, who originally joined the company at Whiting. Dr. G. F. Ordeman, assistant general superintendent at Whiting succeeded Dr. McGill and A. F. Endres, superintendent of the light-oils division at Whiting, succeeded Dr. Odeman.

Montgomery joined Standard Oil Company (Indiana) in 1919 as a chemist at Wood River. After serving as assistant superintendent of the paraffine department, superintendent of the lubricating and paraffine department, and as assistant general superintendent, general superintendent, and manager of the refinery, he was transferred to Whiting as manager in 1942. He was graduated from Mississippi State College and served in the chemical warfare service during World War I.

Dr. McGill joined the company in 1924 as a chemist at Whiting. In 1928 he was transferred as lubricating works assistant to Wood River, where he be-

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# THE MONTH...

came general foreman of the lubricating works and assistant general superintendent and then general superintendent of the refinery before being promoted to manager in 1942. He is an alumnus of the University of Michigan.

Dr. Oderman became associated with the company in 1919 as a chemist at Whiting. After serving as assistant superintendent of the asphalt and acid department he entered the light-oils division where he was successively head of the finishing, crude-still, and pressure-still departments and superintendent of the light-oils division. He was promoted to assistant general superintendent of the refinery in 1943. He is an alumnus of Washington and Lee and Johns Hopkins universities and was a first lieutenant in World War I.

Endres joined the company in 1923 as a chemical engineer at Whiting. He was assigned to the light-oils division as head of the finishing department and after serving also in the crude-still and pressure-still departments and as assistant superintendent of the division was advanced to superintendent of the light-oils division in 1943. He was graduated from the University of Illinois.

## C. A. Johnson Heads California Refiners

C. A. Johnson, Socal Oil & Refining Company, has been elected president of the Independent Refiners' Association of California. D. B. O'Neill is vice president and general manager; W. W. McCallen, McCallen Refining Company, vice president, and J. A. Novelli, Elm Oil Company, secretary-treasurer.

## Northern California Chemical Engineers Elect A. G. Stern

A. George Stern of Westvaco Chlorine Products Company, Newark, California, has ben elected chairman of the Northern California chapter of the American Institute of Chemical Engineers, succeeding C. R. Nelson of Shell Development Company. Other new officers are John R. Callaham, Chemical & Metallurgical Engineering, San Francisco, vice chairman, and Jose A. Samaniego, Shell Development Company, San Francisco, secretary-treasurer. Elected to the executive committee were J. H. Arnold, California Research Corporation, Richmond; P. M. Huemmer, Union Oil Company, Oleum, and B. W. Van Arsdel, United States Department of Agriculture, Albany, California.

Plans are being made for a regional convention to be held at San Jose, California, early in the autumn.

E. F. KINDSVATER

## Neal Rejoins Phillips After Long Army Duty

Hugh A. Neal has rejoined Phillips Petroleum Company as assistant manager of the Perco Division of the chemical products department, at Bartlesville, Oklahoma, after more than five years in military service, from which he was discharged as a lieutenant colonel. Neal entered the Army on September 15, 1940, and trained troops for three years before going overseas in April, 1943. He commanded the artillery of the first parachute combat team going into Sicily, and saw continuous action in Italy until wounded on May 31,. 1944. After hospitalization he was assigned to the field artillery school at Fort Sill, Oklahoma, until his recent release.

## Kindsvater Heads Phillips Chemical Engineering

E. F. Kindsvater has been appointed manager of the chemical engineering department of Phillips Petroleum Company, Bartlesville, Oklahoma, with P. M. Arnold as assistant manager.

Kindsvater has been with the company since 1930, and has been chief engineer since 1940.

Arnold started with the company in 1937 in the research department, and since 1943 has been manager of the process engineering division of the chemical products department.

## McCarthy Plant to Process 85 Million Cubic Feet

Formal opening of the Glenn H. McCarthy gasoline absorption plant, processing 85 million cubic feet daily of casing head and distillate gas, was held March 6. The plant, in Jefferson County near Winnie, Texas, receives 30 million cubic feet of casinghead and 55 million cubic feet daily of distillate gas from four fields in the vicinity — Winnie, Stowell, Pannett, and Fig Ridge. Approximately 40,000 gallons daily of propane, butane, natural gasoline, and diesel fuel is extracted from the casinghead gas, which previously was vented in the field.

E. O. Thompson, Texas railroad commissioner, and Glenn H. McCarthy, head of McCarthy interests, spoke at the opening, which was attended by numerous business and civic leaders who were entertained at a barbecue.

Gas will be supplied by the plant to Jefferson Pipe Line Company and Beaumont Natural Gas Company, McCarthy subsidiaries, and the Neches Butane Company, Texas Public Service Company, and domestic outlets at Central Gardens, Port Neches, and Nederland. Future deliveries will be made to the Du Pont plants in Orange County.

The plant utilizes ten 800-horsepower compressors, two large boilers, two stills, high- and low-pressure absorbers, five towers for product extraction, 15 horizontal storage tanks—total capacity, 525,000 gallons, shipping facilities of a 20-car railroad loading rack and a truck loading rack.

Personnel of the new plant inculde J. J. Sappington, plant superintendent; Leon Dorsey, process engineer, and W. E. Vaughn, chief clerk.

## Natural Gas Liquefaction Plant to Rio Grande Valley

Construction of a plant for converting natural gas into motor fuel and diesel fuel at Brownsville has been announced by Carthage Hydrocol Corporation. Previously the plant, which will cost $15,000,000, had been under consideration for the Carthage field, East Texas. The change in location will permit the use of gas from several fields in the Rio Grande Valley area. The process is a modification of the Fischer-Tropsch, used in Germany for conversion of methane into liquids.

The plant will be a venture sponsored by established companies, among them Chicago Corporation, Forest Oil Corporation, Gulf States Oil Company, La Gloria Corporation, Niagara Shares Corporation, Stone and Webster Engineering Company, The Texas Company and United Gas Corporation.

The plant will process 60,000,000 cubic feet of dry gas daily, from which the

## Trends of Operations and Changes in Stocks

Figures on crude stocks are from Bureau of Mines weekly reports; all others from American Petroleum Institute weekly reports, which are estimates on Bureau of Mines basis.

(All figures in thousands of barrels—add 000)

| Trends in Week Ended: | Crude Oil | | | Gasoline | | Gasoil and Distillate | | Residual Fuel | |
|---|---|---|---|---|---|---|---|---|---|
| | Production Daily | Runs to Stills Daily | Stocks Week End | Production Weekly | Stocks Week End | Production Weekly | Stocks Week End | Production Weekly | Stocks Week End |
| 1945: | | | | | | | | | |
| January 27 | 4,727 | 4,756 | 221,310 | 14,957 | 88,223 | 4,843 | 33,561 | 9,252 | 51,119 |
| February 24 | 4,777 | 4,803 | 219,351 | 15,500 | 95,972 | 4,958 | 28,753 | 9,084 | 46,713 |
| March 31 | 4,781 | 4,677 | 223,782 | 14,644 | 98,758 | 4,548 | 26,889 | 9,184 | 41,745 |
| April 28 | 4,805 | 4,780 | 223,474 | 14,633 | 94,068 | 4,636 | 28,273 | 9,379 | 39,813 |
| May 26 | 4,887 | 4,950 | 222,831 | 15,194 | 89,121 | 4,667 | 29,184 | 9,670 | 38,548 |
| June 30 | 4,903 | 4,999 | 220,781 | 15,546 | 86,472 | 4,910 | 32,213 | 9,077 | 40,488 |
| July 28 | 4,930 | 4,996 | 218,507 | 16,106 | 86,008 | 4,598 | 36,071 | 9,586 | 42,283 |
| August 25 | 4,892 | 4,931 | 211,813 | 15,986 | 84,693 | 4,960 | 39,782 | 9,356 | 46,201 |
| September 29 | 4,357 | 3,812 | 222,387 | 11,913 | 79,552 | 3,940 | 43,689 | 7,047 | 46,853 |
| October 27 | 4,273 | 4,838 | 224,230 | 15,530 | 74,335 | 5,159 | 43,472 | 8,691 | 45,943 |
| November 24 | 4,469 | 4,648 | 219,363 | 15,681 | 83,184 | 4,802 | 45,258 | 8,800 | 47,474 |
| December 29 | 4,474 | 4,729 | 218,918 | 14,546 | 95,205 | 5,055 | 36,651 | 8,765 | 42,447 |
| 1946: | | | | | | | | | |
| January 26 | 4,626 | 4,553 | 220,544 | 13,622 | 107,737 | 5,720 | 29,498 | 8,411 | 39,722 |
| February 23 | 4,714 | 4,595 | 224,762 | 13,175 | 104,709 | 5,728 | 25,398 | 7,913 | 39,290 |

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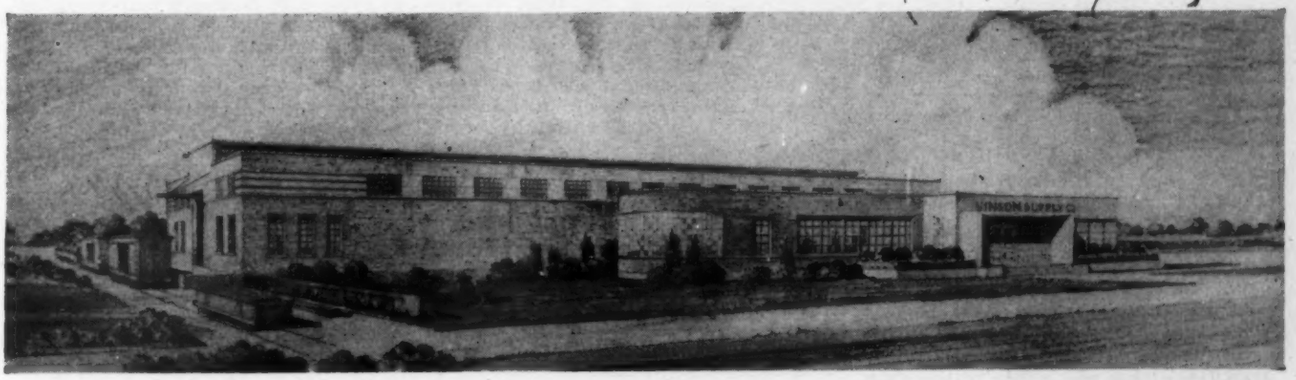


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# THE MONTH . . .

yield is expected to be 6000 barrels of motor fuel and 1000 barrels of diesel fuel.

## Aurora to Build Fluid Catalytic Cracking Unit

Aurora Gasoline Company will add a Fluid type catalytic cracking unit to its refining facilities in Detroit. Its license agreement with Universal Oil Products Company, Chicago, calls for installation and operation of the first UOP design with changed techniques recently announced. The unit will have 4,000 barrels daily capacity.

The company operates a plant of 6,000 barrels daily throughput with thermal capacity of 2400 barrels.

## Continental Oil Company Buys New Mexico Plant

Continental Oil Company has purchased the refining plant of Malco Refineries, Inc., at Artesia, New Mexico. Purchase price was not revealed.

The purchased property adjoins a plant owned and operated for a number of years by Continental Oil Company, and the two will be consolidated and operated as one refinery, it was said. The consolidated plant will have a daily crude charge capacity of 2500 barrels.

## Last Minute Postponement For Oil Agreement Hearing

The Senate foreign relations committee ordered an indefinite postponement of the hearings on the Anglo-American petroleum agreement scheduled for March 4, giving no further reason than that they would have "complicated other matters" probably the handling of the proposed loan to Britain on which hearing is to start March 5.

The postponement was ordered at the request of the State Department which will be occupied with the loan hearings.

## Say $33,000,000 Needed For Science Foundation

Establishment of a National Science Foundation to guide the nation's scientific research has been recommended in reports by subcommittees of the Senate military affairs and commerce committees.

The two subcommittees estimated that it would cost $33,000,000 to get the program under way and $122,000,000 for its conduct in the fifth year of operation. An appropriation of $40,000,000 was recommended in the budget submitted in January.

Arguing that present provision for research is not adequate to meet the needs of the nation, the military affairs subcommittee asserted that "the evidence is clear that America's industrial preeminence will be transitory unless it is strengthened by a firm foundation of basic scientific research, which can be supplied only by a general federal program of the activity in diverse private and public scientific facilities.

The proposed agency would operate no facilities, but would "support science through existing facilities, primarily those of universities, government laboratories, and other non-profit institutions which are recognized to be public centers of research and training."

## PAW Book Reviews Part Played in War

"Petroleum in Peace and War" is the title of a 214-page book issued by Petroleum Administration for War. It consists of papers presented by PAW before the O'Mahoney Special Senate Petroleum Committee.

The book contains a volume of interesting data, and in the event of another war should be the blueprint by which petroleum affairs of such a war will be handled. PAW men who had a part in it are being congratulated on the job they did in preparing the data.

## Engineering Company Moves Office to New York

Project Engineering Company has moved its main engineering design office to 15 Maiden Lane, New York 7, and will continue to maintain operation of fabricating facilities at Long Island City.

The company, recently organized, operates as engineering contractors under the management of Guy M. Barbolini, Fitch B. Jefferies and James P. O'Donnell, who formerly were associated as engineers with The M. W. Kellogg Company. They are now engaged in all phases of design, fabrication, construction and operation of complete projects, especially those related to the operation of modern chemical and petro-chemical units and plants involving new processes and mechanical equipment design.

Presently the company is engaged in the erection of a project, which is part of the reconversion program, for a plant in Michigan and will shortly make test runs on a spray drying unit.

G. M. BARBOLINI

F. B. JEFFERIES

J. P. O'DONNELL

## Brown & Root Forms Unit For Petroleum Construction

Brown & Root, Inc., Houston, has expanded its organization by adding a petroleum and chemical division which offers complete service in engineering and construction of plants for all phases of petroleum and chemical processing. Milton H. Roths is manager of the division, with F. W. Bell, process engineer, and Howard M. Anderson, engineer, as his immediate assistants. Offices for design and engineering and shops for fabrication are located at the Greens Bayou plant of Brown Shipbuilding Company on the Houston Ship Channel, and will be in addition to the yard's shipbuilding, ship repair and drydocking services, and steel fabrication.

Roths recently was released, with rank of commander, from Navy service in the late war, He has had 16 years experience in design, engineering, fabrication and construction of process plants. He graduated from the Naval Academy in 1924, and after two years in the Navy went with C. F. Braun & Company, from 1926 to 1930; with Jackson Engineering Company, Tulsa, 1930 to 1933, and then with Fluor Corporation until 1942 when he was called for active duty in the Navy.

Bell has had 10 years experience as a refinery technologist and in design, engineering and construction of process plants since his graduation in chemical engineering from University of Michigan in 1936. He was with Shell Oil Company from 1936 to 1941, and then with Fluor Corporation until his present connection. He is a member of American Institute of Chemical Engineers.

Anderson has had 20 years experience in the petroleum field since graduation from Kansas State University in 1926. After working with a number of companies in the Mid-Continent, he joined Fluor Corporation as chief draftsman at Kansas City in 1937, and subsequently was project engineer, assistant Mid-Continent manager and assistant chief engineer during the nine years with that company before moving from Los Angeles to his present connection at Houston.

Brown & Root interests, during five years of ship construction at its Houston plant has employed experts in the type of fabrication work required in its added line, and the yard facilities include equipment for shaping, machining and handling the heavy parts involved. The organization has been engaged in general engineering and construction for a number of years.

## Blaize-Armstrong New Houston Engineering Firm

The Blaize-Armstrong Engineering Company, a partnership including R. N. Blaize and J. L. Armstrong, recently was organized to engage in consultation and design, with offices at 902 M. & M. Building, Houston 2. Both Blaize and Armstrong have had more than 12 years' experience in the oil-refining industry, and the company will specialize in the oil-refining, natural-gasoline and allied industries.

Blaize is a chemical engineering graduate of Louisiana State University and has been engaged in research and development, maintenance, design and construction engineering with Lion Oil Company, Ozark Ordnance Works and Eastern States Petroleum Company. He

# THE MONTH...

is an active member of the American Institute of Chemical Engineers.

Armstrong was graduated in civil engineering from Rensselaer Polytechnic Institute, and his experience has been with Phillips Petroleum Company, Lion Oil Company, Dow Chemical Company and Mid-Continent Engineering Company.

## Report Completed on Catalytic Fuel Oils

Catalytic cracked fuel oils are satisfactory for pressurne and wall-type oil burners, but not for the vaporizing pot type, according to the report finally released on tests conducted by the Oil Heat Institute of America and Technical Committee E of the ASTM. The report is based on 21 equipment and 23 equipment control tests conducted in various laboratories over the last year.

The survey was undertaken following complaints of the performance of "cat cracker" distillate in certain burners. More than a year ago PAW, petroleum experts and burner-industry spokesmen engaged in a verbal battle as to the merits of the catalytic products. The tests were undertaken as a result, and some of the findings are:

Seven out of eight laboratories which ran tests on space heaters equipped with vaporizing pot-type burners disapproved the reference oils in all blends.

Six laboratories worked on pot-type furnaces, floor furnaces and conversion vaporizing pot-type burners and found that catalytic products required servicing of equipment every 60 days, and resulted in heavy carbon deposits.

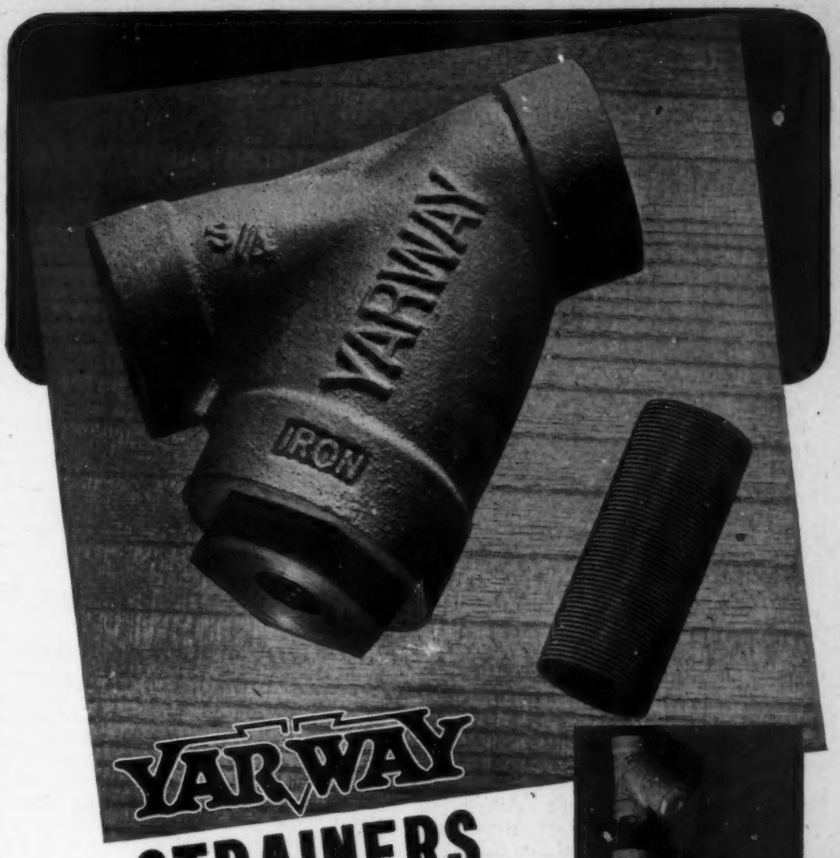
Eleven laboratories studied effects on high-pressure, low-pressure and wall-flame burners. Smoking was reported with catalytic oils in the high-pressure type for five to seven minutes after starting, and carbon collection was noted. In the wall-flame type tests, ignition was found to be slow.

In all cases three disadvantages were reported: excessive carbonization at the oil inlet to the pot, deposits of soot and carbon on secondary surfaces, and reduction in maximum Btu output capacity.

## Laboratory to Be Completed in 1947

Construction has started on buildings for the Standard Oil Company (Indiana) petroleum research laboratories at Hammond, Indiana. Covering two city blocks adjacent to the company's Whiting refinery, the new laboratories will be ready for occupancy early in 1947. Upon completion they will house 420 chemists, chemical engineers, and other scientists, along with 800 technicians and other assistants.

Scheduled for completion in the initial part of the construction are the administration and chemical engineering building, the main research laboratories, and the pilot-plant buildings equipped for work on greases, catalysts, and chemicals. There will be a separate building for research on distillation. The cafeteria, storage facilities, warehouse, and garage will also be finished at this time. New engineering laboratories and the greenhouse for special work on plant sprays will be completed later.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Digest of Recently Issued United States

# Patents Pertaining to Petroleum Refining

Compiled by HEINZ HEINEMANN

## ALKYLATION

**U.S.P. 2,388,918. HF Alkylation Process.** H. O. Iverson to Universal Oil Products Company.

An alkylation process, using a HF catalyst and including fractionation of the alkylation products and purification and regeneration of the catalyst is described.

**U.S.P. 2,389,445. Production of Ethyl Benzene.** W. J. Mattox and W. L. Benedict to Universal Oil Products Company.

Polyethyl benzene and benzene are converted into monoethyl benzene by contacting a mixture containing at least 1 mol of benzene per mol of polyethyl benzene with a catalyst comprising a composite of silica and alumina at above 550° C. and at a space velocity of 0.15 liquid volumes per volume per hour.

**U.S.P. 2,389,550. Catalytic Alkylation Process.** W. E. Ross, S. H. McAllister and J. Anderson to Shell Development Company.

An iso paraffin is alkylated with an olefin in the presence of a liquid mixture of a complex double compound of anhydrous $AlCl_3$ and a hydrogen halide and a metal salt (such as $SbCl_3$) which does not react with $AlCl_3$ at a temperature below 200° C. The mixture must have a melting point below 200° C.

**U.S.P. 2,389,739. Conversion of Hydrocarbons.** E. F. Pevere to The Texas Company.

A molar excess of propane is reacted with ethylene at 175-190° F. and under sufficient pressure to maintain propane in liquid phase. The reaction is carried out in the presence of an $AlCl_3$ catalyst and a hydrogen halide promoter present in a concentration of 4-15 percent by weight.

**U.S.P. 2,390,285. Conversion of Hydrocarbons.** G. B. Zimmerman to Universal Oil Products Company.

Normal and iso butane are subjected to dehydrogenation to form butadiene. The products are separated into hydrogen, and light gases, butadiene and other $C_4$ hydrocarbons. The $C_4$ hydrocarbons containing i-butane, n-butane and butenes are subjected to alkylation, n-butane is removed from the alkylation products and recycled to the dehydrogenation step.

**U.S.P. 2,390,953. Alkylation of Aromatic Hydrocarbons.** S. Mann to The Atlantic Refining Company.

Benzene is reacted at 350-700° F. and under 200-1000 psi pressure with an olefin and a lower aliphatic alcohol in the presence of a phosphoric acid catalyst. The amount of alcohol employed is regulated so as to maintain the partial pressure of water vapor resulting from the alkylation reaction at 40-50 percent of the vapor pressure of the phosphoric acid catalyst at operating conditions.

**U.S.P. 2,391,148. Process for Reacting Propane with Olefins.** F. E. Frey to Phillips Petroleum Company.

Propane is catalytically alkylated with ethylene at 100-250° F. and under sufficient pressure to maintain the reaction mixture in liquid phase. The catalyst consists of HF containing from 2-7 percent of $BF_3$. A propane to ethylene ratio of 2:1 to 10:1 is maintained.

**U.S.P. 2,391,399. Isomerization of Paraffins.** C. G. Dryer to Universal Oil Products Company.

Paraffin hydrocarbons are converted with $AlCl_3$ and HCl under isomerizing conditions and the reaction products are commingled with a nitroparaffin solvent which selectively removes $AlCl_3$ from the hydrocarbon mixture. Isomeric hydrocarbons are separated from unconverted material and contacted with an olefin under alkylation conditions with the $AlCl_3$-nitro paraffin solution, to form antiknock motor fuel.

**U.S.P. 2,391,415. Treatment of Hydrocarbons.** A. V. Grosse and C. B. Linn to Universal Oil Products Company.

Isoparaffinic hydrocarbons are alkylated with an olefin polymer under conditions which tend to depolymerize the polymer and alkylate the isoparaffin with olefinic fragments thereof. A catalyst consisting of nickel, HF and $BF_3$ is employed.

**U.S.P. 2,392,466. Production of Styrene.** W. I. Deuton and C. H. Schlesman to Socony-Vacuum Oil Company.

Styrene is obtained directly from benzene and ethylene by passing them in the vapor phase together with an oxygen-containing gas such as air through a heated zone at 600-1100° F. and under 10-200atm. pressure for a period of 1 second to 1 minute. Enough oxygen-containing gas is used to react with at least part of the hydrogen of the benzene and ethylene.

**U.S.P. 2,392,962. Alkylation Process.** A. J. Abrams and C. S. Kuhn, Jr. to Socony-Vacuum Oil Company.

The reaction time of an alkylation reaction between an isoparaffin, olefin and HF catalyst is controlled by diluting the hydrofluoric acid with water below its effective concentration and thus instantly stopping the reaction.

**U.S.P. 2,393,152. Alkylation.** C. Ellis to Standard Oil Development Company.

Isoparaffins and mono-olefins are reacted under alkylating conditions in the presence of a sulfuric acid catalyst containing as promoters wetting agents consisting of high-molecular-weight N-acyl taurines and sulfuric acid esters of high-molecular-weight aliphatic monohydric alcohols.

## ISOMERIZATION

**U.S.P. 2,390,883. Isomerization of Saturated Hydrocarbons.** J. O. Iverson to Universal Oil Products Company.

Hydrogen and HCl, separated from the effluent gas of an isomerization zone are contacted with a liquid catalyst solution comprising hydrocarbons, $AlCl_3$ and such an amount of hydrogen as is soluble therein. The HCl is dissolved in the solution and thus separated from hydrogen.

**U.S.P. 2,391,189. Isomerization Process.** S. F. Perry to Standard Oil Development Company.

Normal butane containing small amounts of sulfur-type impurities is passed in admixture with promotional amounts of HCl through a bed of $AlCl_3$ sorbed on a porous carrier. The reaction zone is kept at 250-325° F. and under 150-300 psi pressure. The effluent is stripped and a mixture comprising HCl, hydrocarbons lighter than butane and $H_2S$ is removed overhead and passed into contact with $AlCl_3$ to remove $H_2S$ therefrom. HCl is recycled to the isomerization reaction.

**U.S.P. 2,391,775. Processing of Hydrocarbons.** E. W. M. Fawcett and E. S. Narracott to Anglo-Iranian Oil Company.

Paraffin hydrocarbons are isomerized at 50-200° C. and 1-10 atm. pressure in the presence of an $AlCl_3$ catalyst, and 5 percent or less (on the basis of hydrocarbon feed) of HCl and traces of water. The molar concentration ratio of water to HCl present in the feed is within the limits of 0.0001-0.01.

**U.S.P. 2,392,791. Catalytic Isomerization Process.** W. O. Webber to Standard Oil Development Company.

A mechanical arrangement for carrying out the isomerization of a n-paraffinic hydrocarbon in the presence of a catalyst and a gaseous promoter is described.

**U.S.P. 2,392,924. Production of Isobutane.** V. Ipatieff and H. Pines to Universal Oil Products Company.

N-butane is converted to i-butane in the presence of anhydrous stannic chloride and hydrogen chloride at about 150° C. and under 10-50 atm. pressure.

**U.S.P. 2,393,041. Process for the Isomerization of Paraffin Hydrocarbons.** B. S. Greenfelder to Shell Development Company.

Paraffin hydrocarbons having from 4-6 C atoms per molecule can be isomerized in admixture with hydrogen at 400-540° C. and under 10-100 atm. pressure in the presence of a heavy metal sulfide catalyst such as molybdenum sulfide which may contain a minor amount of a metal of the iron group. The beneficial effect of the hydrogen is not a function of its partial pressure but is a function of the ratio of partial pressures of hy-

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**MAXIMUM REFLUX RATIO FOR MULTICOMPONENT DISTILLATION**
F. D. Mayfield and J. A. May, Styrene Division, Dow Chemical Co.

**RAPID TEST FOR ISOBUTANE FRACTIONS IN ABSORBER RESIDUE CASES**
Staff of Standard Oil Company, California

**EXTENSION OF API HYDROMETER TABLES**
Dr. John Griswold, University of Texas

**DOUBLE PROCESSING FOR RICH AND LEAN GASES**
Staff contribution

**FUNDAMENTALS OF EVAPORATION OF LIGHT HYDROCARBONS**
H. C. Boardman, Chicago Bridge & Iron Co.

**CONTROL OF FRACTIONATING TOWERS**
D. M. Boyd, Jr., Monsanto Chemical Co.

**DESIGN OF COMPRESSOR CYLINDERS**
H. A. Gehres, Cooper-Bessemer Corp.

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drogen to hydrocarbons. This ratio should vary between 1.5:1 and 8.6:1.

**U.S.P. 2,393,051. Isomerization of Pentane.** F. M. McMillan and H. A. Cheney to Shell Development Company.

N-pentane in admixture with i-butane is contacted in vapor phase at a temperature not exceeding 100° C. with a catalyst comprising an aluminum halide and a porous support material. The initial molar ratio of i-butane to n-pentane of at least 4:1 is reduced to a minimum of 2:1 as catalyst activity declines.

**U.S.P. 2,393,104. Isomerization of Normally Liquid Paraffins.** A. V. Grosse and H. Pines to Universal Oil Products Company.

N-paraffin hydrocarbons contained in hydrocarbon mixtures are more efficiently isomerized in the presence of a Friedel-Crafts catalyst and a hydrogen halide promoter while in admixture with a non-olefinic hydrocarbon than in its absence.

**U.S.P. 2,393,357. Production of Isoparaffins.** E. W. M. Fawcett and E. S. Narracott to Anglo-Iranian Oil Company.

N-butane is isomerized in the presence of an aluminum chlorobromide catalyst and of 1 percent by volume of a hydrogen chloride promoter. The reaction temperature is 110° C. A good catalyst consists of alumina gel, dried at 450° C., mixed with 6 percent aluminum dichlorobromide.

## HYDROGENATION, DEHYDROGENATION, AROMATIZATION

**U.S.P. 2,377,116. Hydrogenation Over Sulfur-Sensitive Catalysts.** A. Voorhies, Jr. and E. A. Stahly to Standard Catalytic Company.

The relation between the sulfur content of the feed stock and the quantity of hydrogen which must accompany the oil during the reaction in order to avoid substantial sulphiding and deactivation of the sulfur-sensitive catalyst is established.

**U.S.P. 2,388,937. Treatment of Hydrocarbon Oils.** L. Schmerling and V. N. Ipatieff to Universal Oil Products Company.

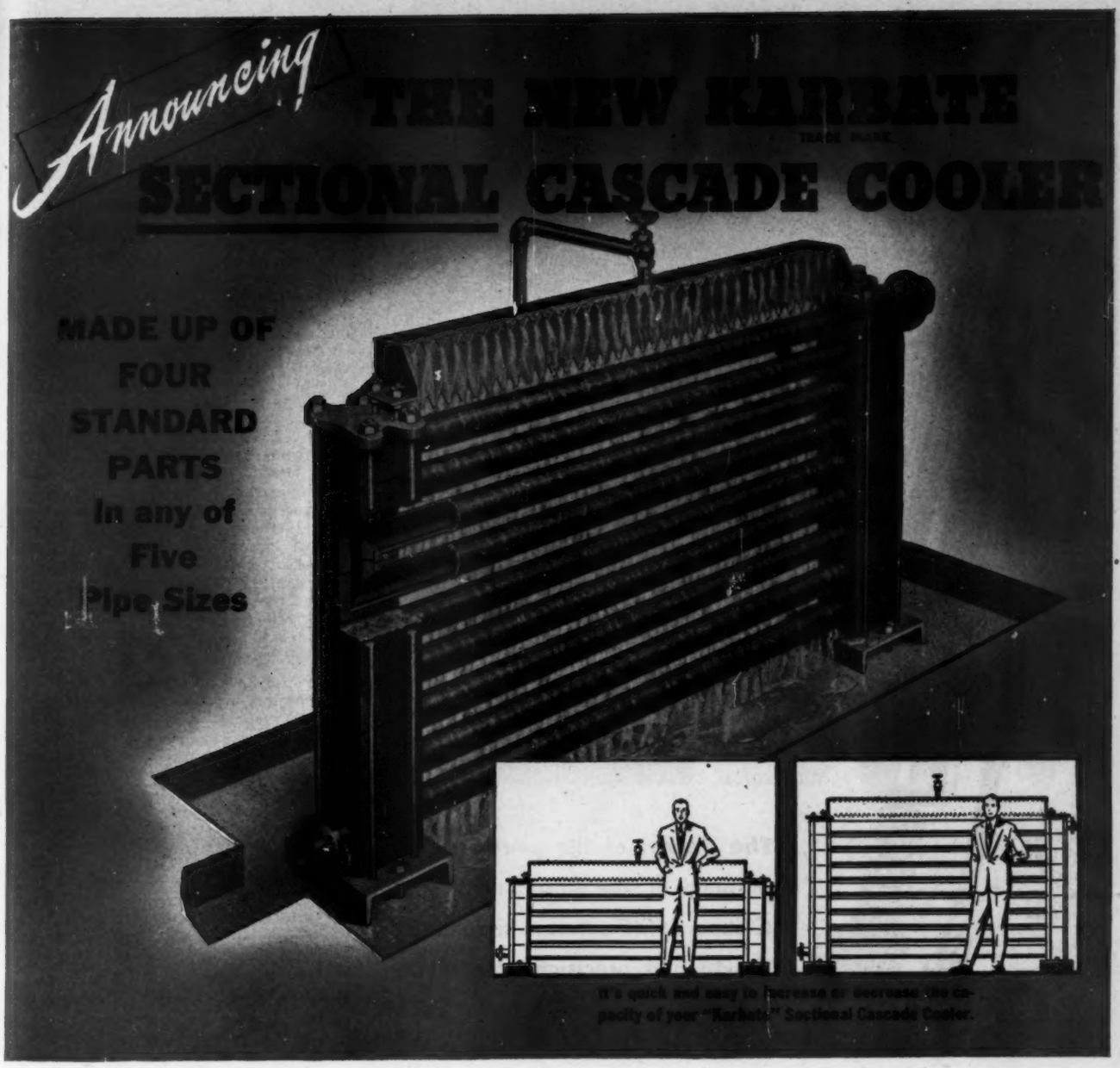
Hydrocarbonaceous materials are hydrogenated at elevated temperatures and pressure and in the presence of hydrogen in contact with granular catalyst comprising essentially composites of zinc chloride and aluminum oxide.

**U.S.P. 2,392,330. Process for the Production of Toluol.** G. E. Liedholm and F. M. McMillan to Shell Development Company.

Toluene is obtained from a naphthenic straight-run gasoline fraction boiling between 190 and 230° F. by fractionally distilling it into two fractions boiling be- 190 and 200, and 200 and 230° F., contacting the first fraction under isomerization conditions with an $AlCl_3$ catalyst, recombining it with the second fraction and subjecting the mixture to catalytic dehydrogenation.

**U.S.P. 2,392,398. Process for the Production of Toluol.** F. M. McMillan and G. E. Liedholm to Shell Development Company.

A naphthenic gasoline fraction boiling between 190 and 218° F. is isomerized with an $AlCl_3$ catalyst. The isomerizate

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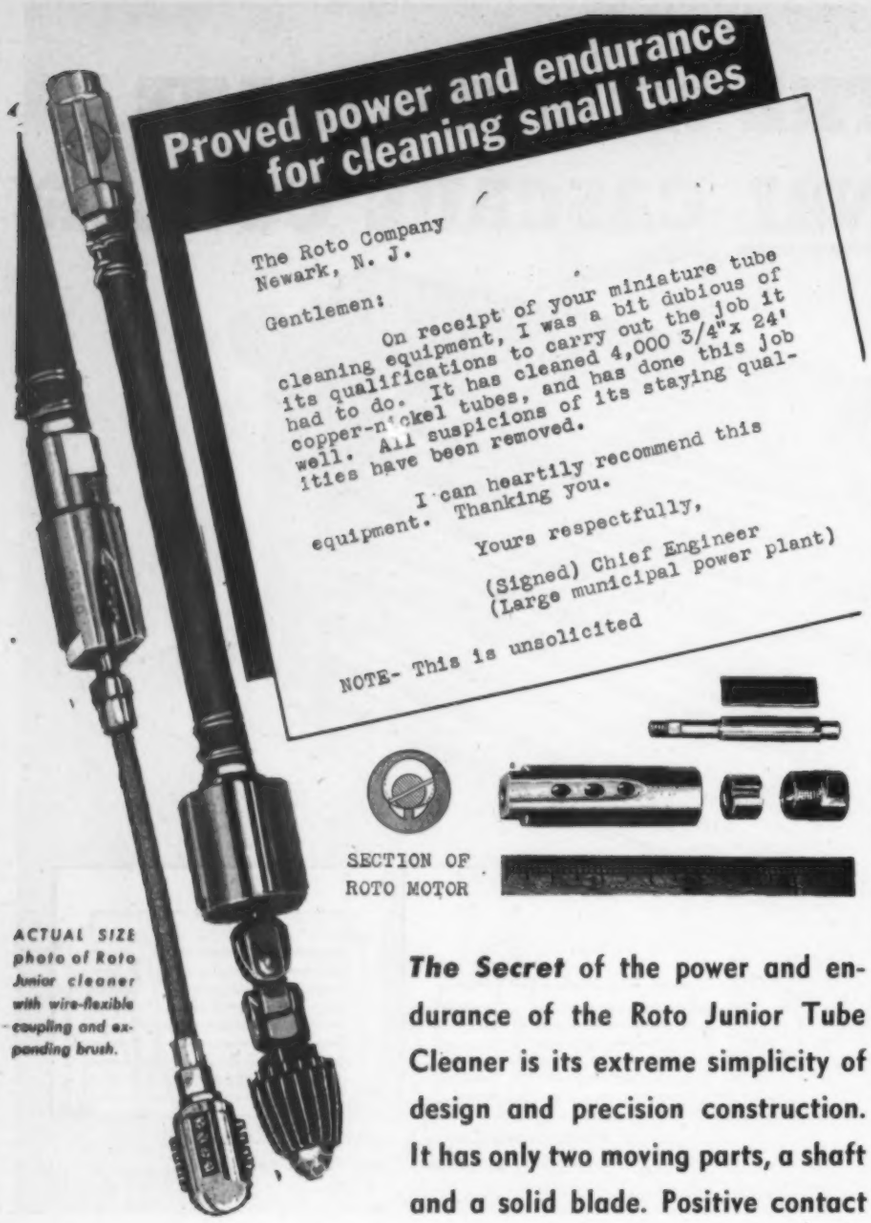
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is fractionated into a lower-boiling fraction (190-210°) and a higher-boiling fraction. The former is again isomerized and the product fractionated. The higher-boiling fractions of the two distillations are combined and dehydrogenated. Substantially pure toluene is recovered from the dehydrogenation product.

**U.S.P. 2,392,749. Production of Aromatic Hydrocarbons from Petroleum.** E. R. Lewis and C. L. Read to Standard Catalytic Company.

Instead of hydroforming a 200-250° F. fraction of a virgin naphtha, higher-boiling fraction so the same naphtha are first subjected to destructive hydrogenation and a 200-250° fraction of the product of destructive hydrogenation is then subjected to hydroforming. The toluene content of the final product is doubled by the latter procedure.

**U.S.P. 2,392,750. Dehydrogenation of Hydrocarbons.** N. F. Linn to Standard Oil Development Company.

Hydrocarbons to be dehydrogenated are passed in admixture with steam over a catalyst containing 50-97 weight percent of zirconium oxide, 30-50 percent of ferric oxide 1-15 percent of copper oxide and 0.5-15 percent of water. A reaction temperature of 1100 - 1300° F., space velocity of 100-1000 vol./vol./hr. and steam : hydrocarbon ratio of between 8:1 and 4:1 are employed.

**U.S.P. 2,392,960. Process for the Dehydrogenation of Alkenylcyclohexenes.** C. W. Watson to The Texas Company.

A cyclic process for the production of styrene from a butane-butene-butadiene fraction is described which comprises subjecting the fraction to a temperature of 650-950° F. under superatmospheric pressure to effect dimerization of butadiene, subjecting the resulting mixture to a temperature of 2100-1400° F. during contact with a dehydrogenation catalyst comprising chromium or molybdenum oxide to effect dehydrogenation of butadiene dimer to styrene, and dehydrogenation of $C_4$ components of the mixture to butadiene.

**U.S.P. 2,393,537. Catalytic Dehydrogenation.** H. C. Huffman to Union Oil Company of California.

Dehydrogenation catalysts are prepared by adsorbing on granular alumina aqueous solutions of salts of beryllium and salts of metals selected from the left-hand column of the sixth group of the periodic table, followed by reduction of the salts to the corresponding oxides. A good catalyst may comprise 90 percent alumina, 5 percent chromia and 5 percent beryllia.

**U.S.P. 2,393,565. Production of Aviation Gasoline.** J. M. Powers to Standard Oil Development Company.

Naphthenes, cyclo-olefins and aromatics are formed from $C_6$ and $C_4$ paraffins at 1000-1100° F. and under 1500-2500 psi pressure. A fraction boiling in the range of 100-175° F. is hydrogenated and saturated ring hydrocarbons are obtained.

## POLYMERIZATION, CONVERSION

**U.S.P. 2,388,076. Process for Stabilizing Petroleum Oil.** G. E. Phillips to Standard Oil Development Company.

An oil containing diolefins is stabilized by the polymerization of the diolefins by passage over an active clay bed at

**PERHAPS SOME DAY *you* MAY BE IN A POSITION TO WRITE A LETTER LIKE THIS**

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If you have a spare copy of the 1945-46 edition, we should very much appreciate having it. Please send it to my attention.

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Chief Engineer

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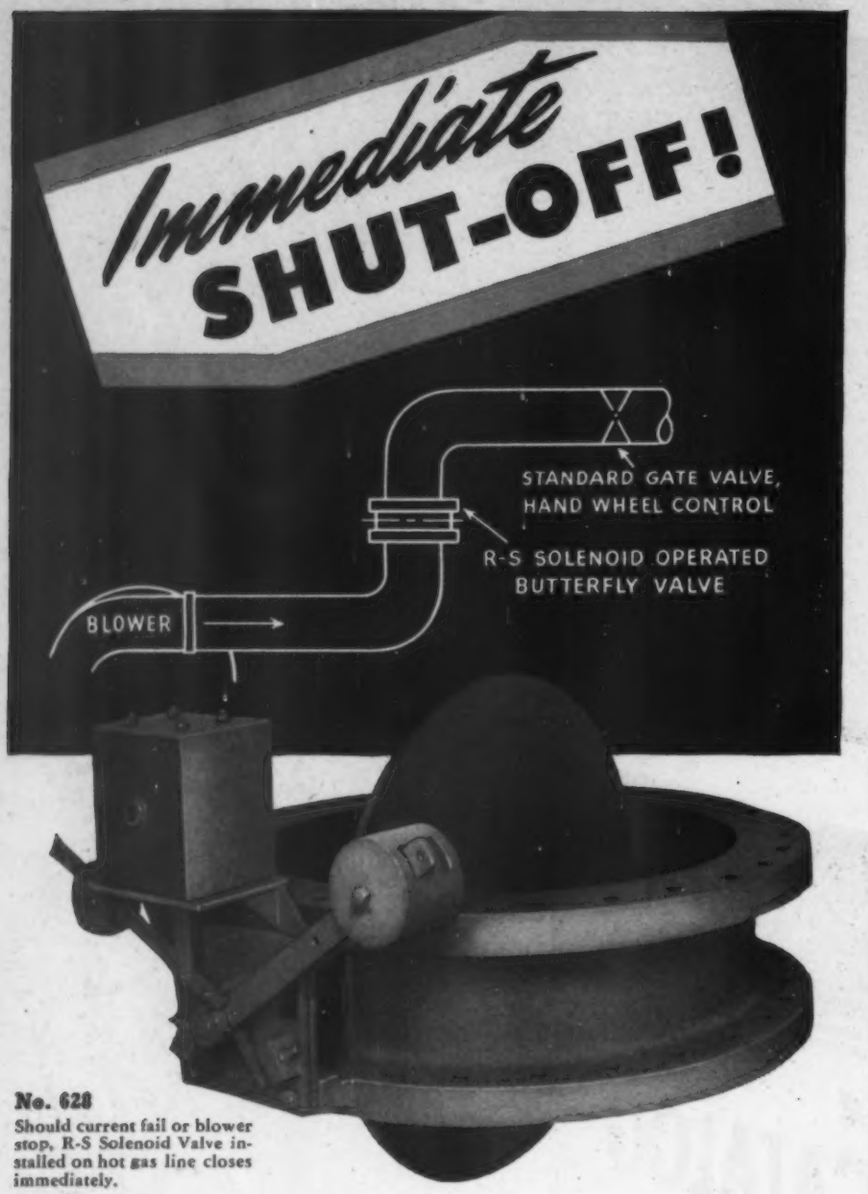


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temperatures as high as 300° F. The temperature within the reaction zone is controlled by introducing water in an amount of not more than 1 percent of the feed at a plurality of points.

**U.S.P. 2,392,000. Polymerization of Hydrocarbons.** C. J. Randolph, Jr. and N. B. Haskell to The Texas Company.

Hydrocarbon vapor consisting mostly of propylene is polymerized in the presence of a catalyst of the approximate composition 100 $SiO_2$ : 2 $Al_2O_3$ : 12 $ZrO_2$ at 425-560° F. The catalyst is left on stream for 2 or 3 hours and for not more than 5 hours before regeneration. The reaction product is relatively free from aromatics and consists mostly of saturates.

**U.S.P. 2,392,284. Catalytic Conversions.** W. P. Gage to Shell Development Company.

In a hydrocarbon conversion process wherein hydrocarbons are converted by passage in admixture with a hydrogen halide at conversion conditions through a catalyst-containing reaction zone, reaction products are passed through an accumulating zone to a fractionating zone, fixed gases comprising hydrogen halide are eliminated from the accumulating zone, and a normally gaseous fraction comprising hydrogen halide is separated from a liquid fraction comprising hydrocarbons in the fractionating zone, the combination of steps which comprises recycling a part of said gaseous fraction to the reaction zone, combining the remainder of said gaseous fraction with the effluence from the reaction zone, bringing said fixed gases into intimate contact with an absorbing medium in an absorber thereby dissolving hydrogen halide in said absorbing medium, passing a part of said liquid fraction to the absorber as the absorbing medium, combining the enriched absorbing medium with the effluence from the reaction zone, cooling the resulting mixed stream to a temperature sufficiently low to dissolve at least a substantial part of the hydrogen halide present in the hydrocarbons, passing the cooled stream to the accumulating zone, and passing liquid comprising dissolved hydrogen halide from the accumulating zone to the fractionating zone.

**U.S.P. 2,392,454. Preparation of Isobutane and Ethyl Chloride.** D. C. Bond and N. B. Russell to The Pure Oil Company.

Hydrogen chloride and ethylene in the ratio of 0.15-0.5 volumes of HCl per volume of ethylene are contacted with a catalyst comprising $AlCl_3$ supported on activated alumina. A reaction temperature of 300-350° F. and a space velocity of 5-30 are employed. The reaction product consists largely of i-butane and ethyl chloride.

**U.S.P. 2,393,569. Catalytic Hydrocarbon Conversion Process.** W. E. Ross and G. J. Carlson to Shell Development Company.

In a catalytic hydrocarbon conversion process wherein hydrocarbons are converted by contact with a fluid catalyst melt comprising antimony trichloride and aluminum chloride at conversion conditions in a conversion zone, and a liquid fraction comprising entrained antimony trichloride is separated from reaction products, the steps which comprise withdrawing a portion of at least partly spent catalyst from said conver-

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sion zone, scrubbing said portion of catalyst with the hydrocarbons to be converted in a scrubbing zone, thereby separating catalyst components by solution from a fluid catalyst residue consisting essentially of aluminum chloride-hydrocarbon complexes, passing hydrocarbons comprising dissolved catalyst components from the scrubbing zone to the conversion zone, passing said fluid catalyst residue from said scrubbing zone into a distillation zone, maintaining a temperature sufficiently high in said distillation zone to convert said catalyst residue into vapors comprising aluminum chloride and a coky residue, contacting said vapors with said liquid fraction comprising entrained antimony trichloride separated from reaction products thereby dissolving aluminum chloride in said liquid fraction, and passing the resulting liquid comprising antimony trichloride and dissolved aluminum chloride to said conversion zone.

## CATALYST PREPARATION ACTIVATION AND REGENERATION

**U.S.P. 2,391,944. Conversion of Hydrocarbon Oils.** L. E. Carlsmith to Standard Oil Development Company.

Valuable constituents are removed from spent catalysts prior to regeneration by stripping the catalyst with a large volume of steam which would be objectionable if allowed to pass into the regeneration zone and thereafter subjecting the catalyst to a second stripping treatment with a relatively small volume of gas which is not objectionable in the regeneration zone.

**U.S.P. 2,392,248. Hydrocarbon Conversion.** E. T. Laying and S. Goldwasser to The M. W. Kellogg Company.

A dehydrogenation and reforming catalyst is described which consists of a major proportion of dehydrated alumina gel, a minor proportion of molybdenum oxide or chromium oxide and a minor proportion of an activating and stabilizing ingredient comprising alumina in the form of a residue of a thermally-decomposed aluminum salt.

**U.S.P. 2,392,738. Process of Aromatizing Hydrocarbons.** C. H. Holder and A. B. Welty, Jr. to Standard Oil Development Company.

A hydroforming catalyst is described which consists of a catalyst base comprising zinc oxide and alumina combined to form a spinel structure and of cobalt molybdate as active component.

## REFINING

**U.S.P. 2,390,536. Separation of Hydrocarbon Gases.** E. J. Houdry and R. W. Blue to Houdry Process Corporation.

Hydrocarbon mixtures of at least two gases one of which is more strongly adsorbed by an adsorbent such as silica gel than the other are separated by passage over an adsorbent material. When the composition of the effluent gas approaches that of the charge the reaction is discontinued and gas enriched in one component is desorbed from the adsorbent.

**U.S.P. 2,390,988. Process of Deleading Gasoline.** G. Calingaert, H. Soroos and H. Shapiro to the United States of America.

Gasoline is deleaded by adding an excess of stannic chloride to gasoline con-

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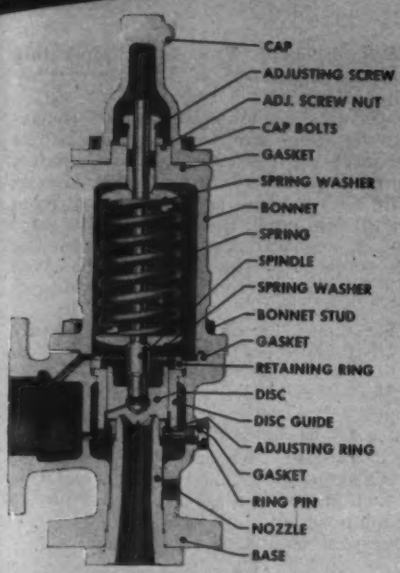
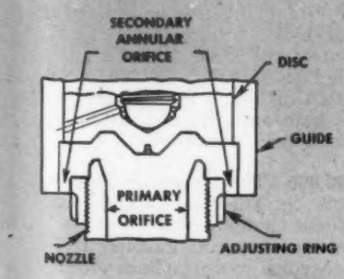



Consolidated Safety Relief Valve which functions with pop action when relieving gases, and as a relief valve when relieving liquids. Note the outside bevel seat which assures tightness.




The secondary annular orifice controls the high-lift pop action of Consolidated Safety Valves. This fixed area cannot be changed.

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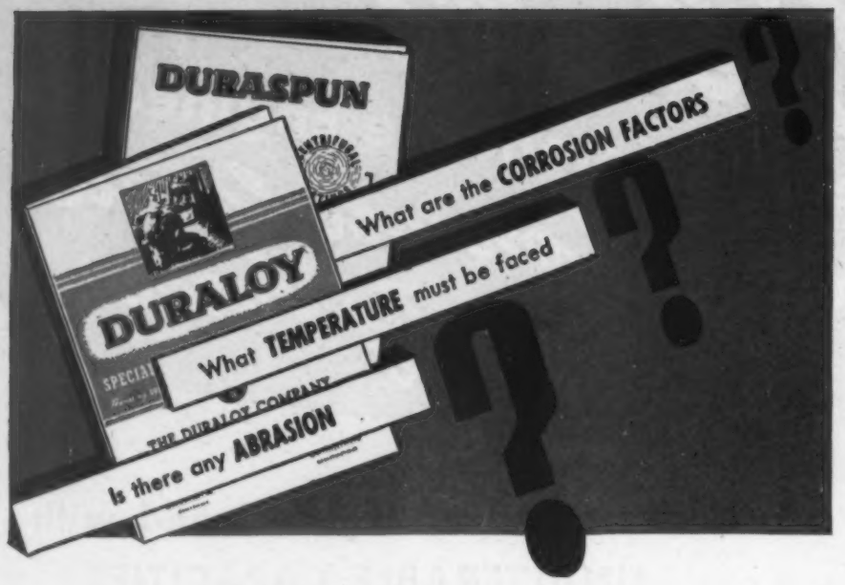


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taining tetraethyl lead, removing the resultant precipitate and washing with water to remove unreacted stannic chloride.

**U.S.P. 2,391,091. Refining Petroleum Oils.** A. P. Hewlett and G. E. Phillips to Standard Oil Development Company.

Straight-run gasoline is contacted in the vapor phase at 780-850° F. with bauxite. Objectionable sulfur compounds are removed and a molecular rearrangement is secured with substantially no cracking of the oil occurring and with less than 1 percent of gas being formed.

**U.S.P. 2,391,149. Process for Treating Hydrocarbons Containing Organically Combined Fluorine.** F. E. Frey to Phillips Petroleum Company.

The removal of F ion from hydrocarbon materials containing the same is accomplished by treatment with alumina impregnated with alkali hydroxide or alkali-metal salt. The treatment is carried out at 150-550° F.

**U.S.P. 2,392,277. Process of Separating Hydrocarbons.** C. E. Welling to Phillips Petroleum Company.

Mixtures of olefins and paraffins having the same number of carbon atoms per molecule are separated by intimately contacting the mixture in the gaseous state with a liquid solvent selected from the group consisting of levulinic acid and its lower aliphatic alkyl esters. The unsaturated hydrocarbons are dissolved and later recovered from the solution.

**U.S.P. 2,392,739. Segregation of Olefins.** H. T. Horeczy and E. F. Wadley to Standard Oil Development Company.

Mixtures of olefins and paraffins are contacted with an iodine solution in a mutual solvent for iodine and aliphatic hydrocarbons. Absorbed hydrocarbons are driven off by heating. The solution is then heated to a higher temperature to decompose alkyl di-iodides for the recovery of olefins.

**U.S.P. 2,392,846. Method for the Removal of Tetraehtyl Lead from Hydrocarbons.** M. Friedman to the United States of America.

In order to remove tetraethyl lead from leaded hydrocarbon fuel, a sufficient amount of anhydrous stannic chloride is added to and agitated with the fuel. Activated carbon is then added, the mixture is again agitated and, after decanting clear liquid, filtered.

**U.S.P. 2,392,896. Treatment of Hydrocarbons.** S. S. Allender to Universal Oil Products Company.

A process for producing gasoline from saturated gaseous hydrocarbons occurring in natural and casinghead gases.

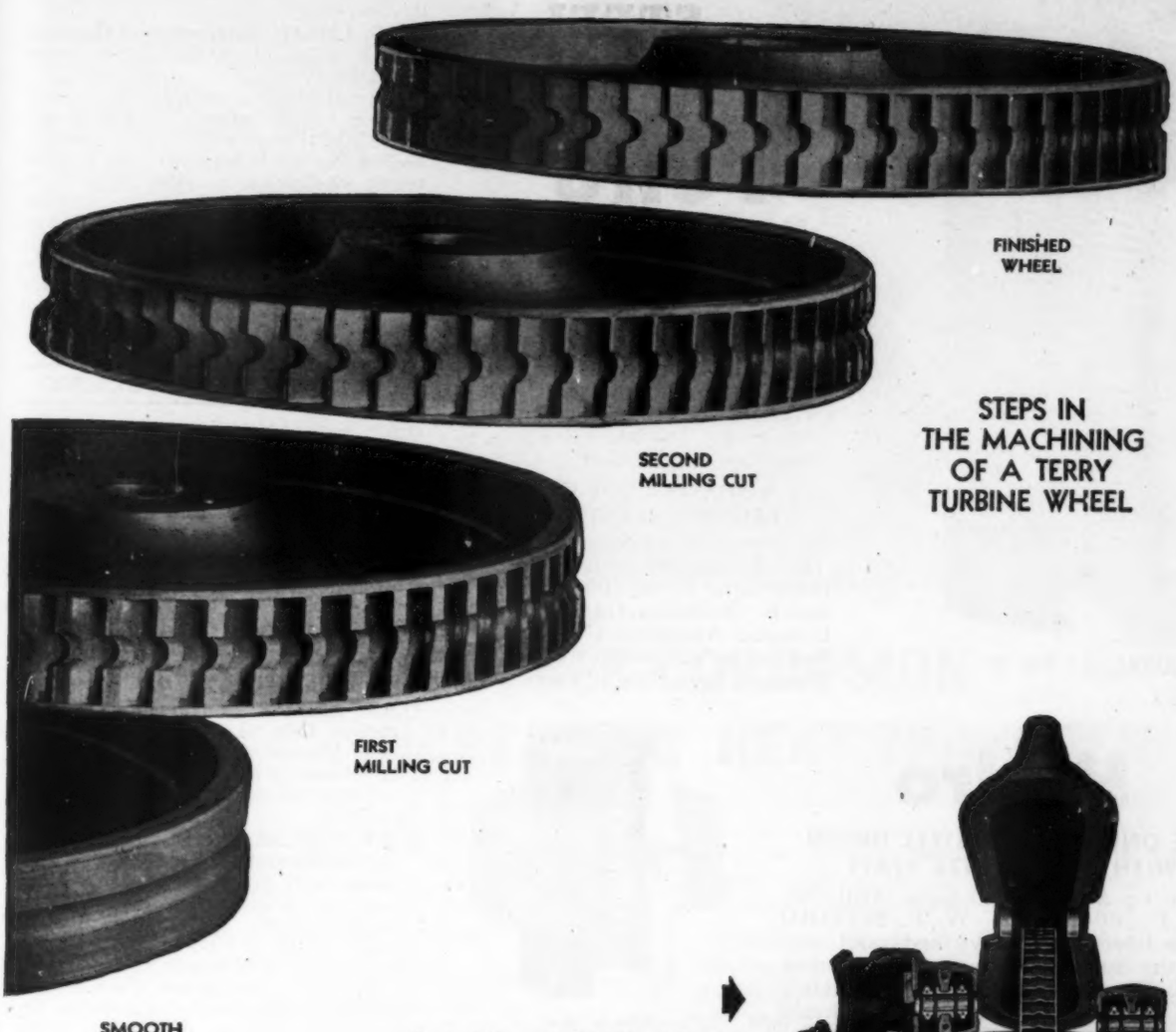
**U.S.P. 2,392,915. Treatment of Hydrocarbons.** A. V. Grosse and C. B. Linn to Universal Oil Products Company.

Hydrocarbons produced by catalytic alkylation in the presence of an active fluoride catalyst are treated by contacting them with activated carbon in the presence of small amounts of HF at 20-150° C. Dehydrofluorinated hydrocarbons are recovered.

**U.S.P. 2,393,154. Petroleum Products.** J. L. Franklin to Standard Oil Development Company.

The action of acidic compounds present in alkylates after water and/or caustic washing which tends to corrode

# Wheel Turbine



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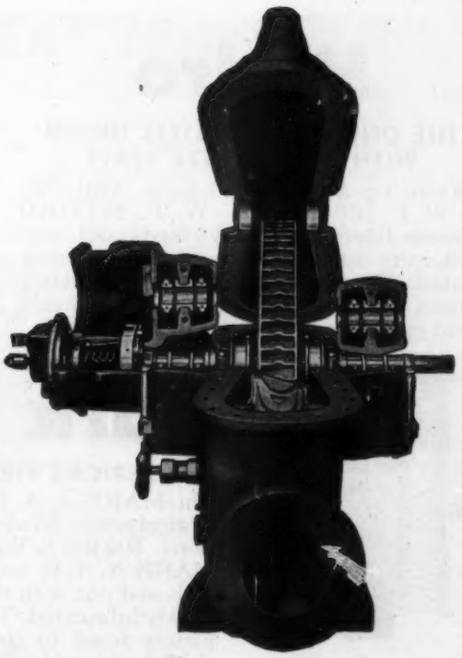

Typical Terry Solid Wheel Turbine with cover and bearing caps raised. Note the ruggedness and the accessibility of all parts. Units of this type are used to drive boiler feed pumps, generators, and all types of power plant equipment. Built in sizes from 5 H.P. to 2000 H.P.

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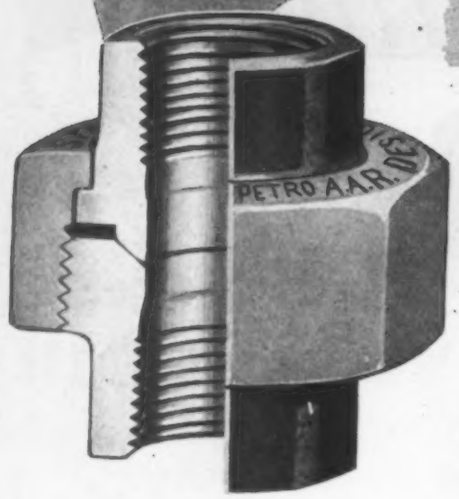


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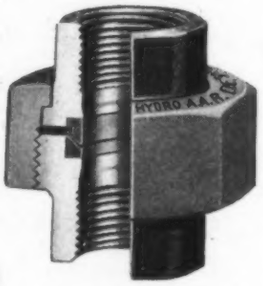
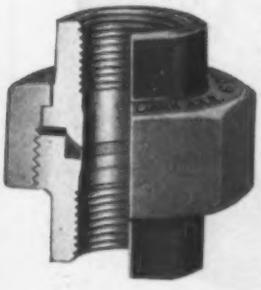



SIZES ¼" TO 3"



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distillation equipment is inhibited by adding a small quantity of an amine to the neutralized water-washed alkylate prior to distillation.

**U.S.P. 2,393,531. Chemical Process.** J. L. Hart to Standard Oil Development Company.

Corrosion of distillation equipment during the distillation of raw alkylate from a sulfuric acid alkylation reaction is prevented by adding between 0.005 and 0.1 percent by volume of phenol to the alkylate.

**U.S.P. 2,393,476. Sweetening of Gasoline.** H. E. Messmore to Phillips Petroleum Company.

Sour distillate is separated into two fraction, boiling between 250-350° F. and above 350° F. respectively. The higher-boiling fraction is subjected to a conventional sweetening process, whereby a major proportion of the mercaptans therein are sweetened. The low-boiling fraction is then mixed with the partially sweetened high-boiling fraction and the mixture is subjected to a secondary sweetening step to yield an effluent which is negative to the doctor test.

## CRACKING AND REFORMING

**U.S.P. 2,392,773. Treating Hydrocarbons.** J. R. Schonberg and H. J. Moyer to Standard Oil Development Company.

Better yields of high-quality gasoline are secured from catalytic feed stocks in which the degree of unsaturation and cyclization is high than from stocks in which paraffin hydrocarbons predominate. Gasoil is subjected to thermal treatment at 825-1000° F. and under 1-1000 psi pressure for about 2 minutes to increase its naphthenicity. The recovered gasoil is then catalytically cracked.

**U.S.P. 2,393,028. Conversion of Hydrocarbon Oils.** G. S. Dunham to Socony-Vacuum Oil Company.

A method of preparing a charge stock for vapor-phase catalytic cracking is described.

**U.S.P. 2,393,288. Process for the Catalytic Reforming of Hydrocarbon Mixtures.** A. C. Byrns to Union Oil Company of California.

Hydrocarbons are catalytically reformed at 500 to 1000° F. by contact in the presence of hydrogen with a catalyst comprising a compound selected from the class consisting of chromites and molybdates of iron, cobalt, and nickel precipitated on a previously undried hydrous metal oxide gel. Conversion of non-hydrocarbons such as organic sulfur compounds, phenols and nitrogen bases into readily removable types of compounds is effected.

## ALKYLATION

**U.S.P. 2,387,948. Alkylation of Aromatic Hydrocarbons.** R. E. Schaad to Universal Oil Products Company.

Aromatic hydrocarbons and olefins are alkylated at 200-450° C. and under 1-100 atm. pressure with a catalyst the active component of which comprises magnesium or zinc acid phosphate.

**U.S.P. 2,338,007. Alkylation of Benzene.** W. A. Pardee and B. F. Dodge to Gulf Research and Development Company.

The vapor phase alkylation of benzene and ethylene is effectively promoted by a catalyst formed of sodium-aluminum

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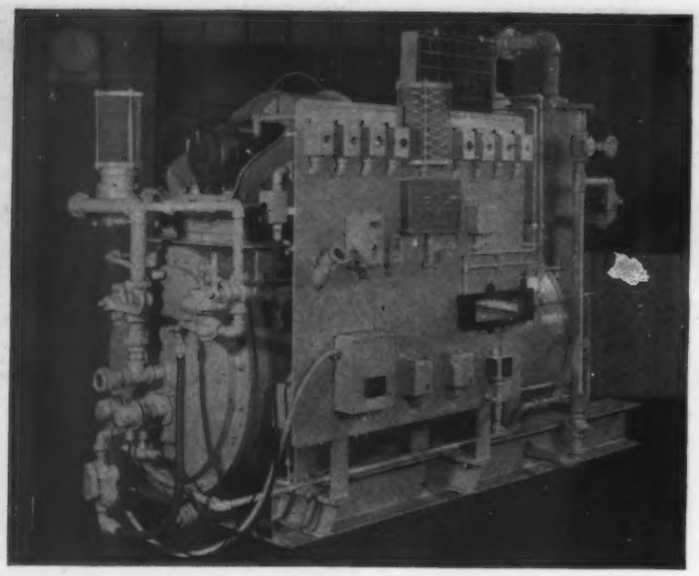

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chloride disposed on a suitable inert support of large surface area. Temperatures of 80-400° C. and a pressure of at least 50 psi are employed.

**U.S.P. 2,388,758. Process of Making Primary Alkylated Benzenes and Naphthalenes.** E. J. Mills, Jr. to Carbide and Carbon Chemicals Company.

Polyethyl benzenes contained in the reaction product from the alkylation of benzene with ethylene in the presence of an $AlCl_3$ catalyst are converted to ethyl benzene by reaction with unconverted benzene. The reaction product is heated in the liquid phase and in the absence of ethylene to 50-150° C.

## HEAVY OILS AND WAXES

**U.S.P. 2,392,252. Lubricant.** R. L. May to Sinclair Refining Company.

To a lub oil is added an effective proportion (0.05-2 percent) of an oxidation inhibitor consisting of the reaction product of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

**U.S.P. 2,392,468. Organic Phosphorous Lubricant Composition.** E. F. Engelke to Cities Service Oil Company.

An improved oil composition is claimed comprising a relatively large proportion of a lubricating oil and a relatively small proportion of an added agent comprising of cresol ester of phosphorus containing an abietate substitutent directly attached to the phosphorus of the ester with a phosphorus-carbon linkage.

**U.S.P. 2,392,570. Method for Improving the Cetane Value of a Hydrocarbon Fuel Oil.** H. G. Oberger and D. E. Badertscher to Socony-Vacuum Oil Company.

Aromatic constituents of the fuel oil are converted to resinous materials by treatment of the oil with an aliphatic aldehyde and hydrogen fluoride. The resinous products are separated from a fuel oil of improved octane number.

**U.S.P. 2,392,610. Diesel Fuel.** T. T. Noland and E. M. Nygaard to Socony-Vacuum Oil Company.

The ignition quality of diesel fuels is improved by incorporation therein of minor proportions of halogen- and nitrogen-containing reaction products obtained by the reaction of nitrous acid and halogen substituted nitro paraffins having a halogen atom, and a nitro group attached to the same carbon atom.

**U.S.P. 2,392,611. Diesel Fuel.** E. M. Nygaard, J. H. McCracken and N. H. Hamilton to Socony-Vacuum Oil Company.

A diesel fuel of improved ignition quality is claimed comprising a minor proportion of a compound containing

$$T-N=N-\underset{|}{\overset{|}{C}}-NO_2$$

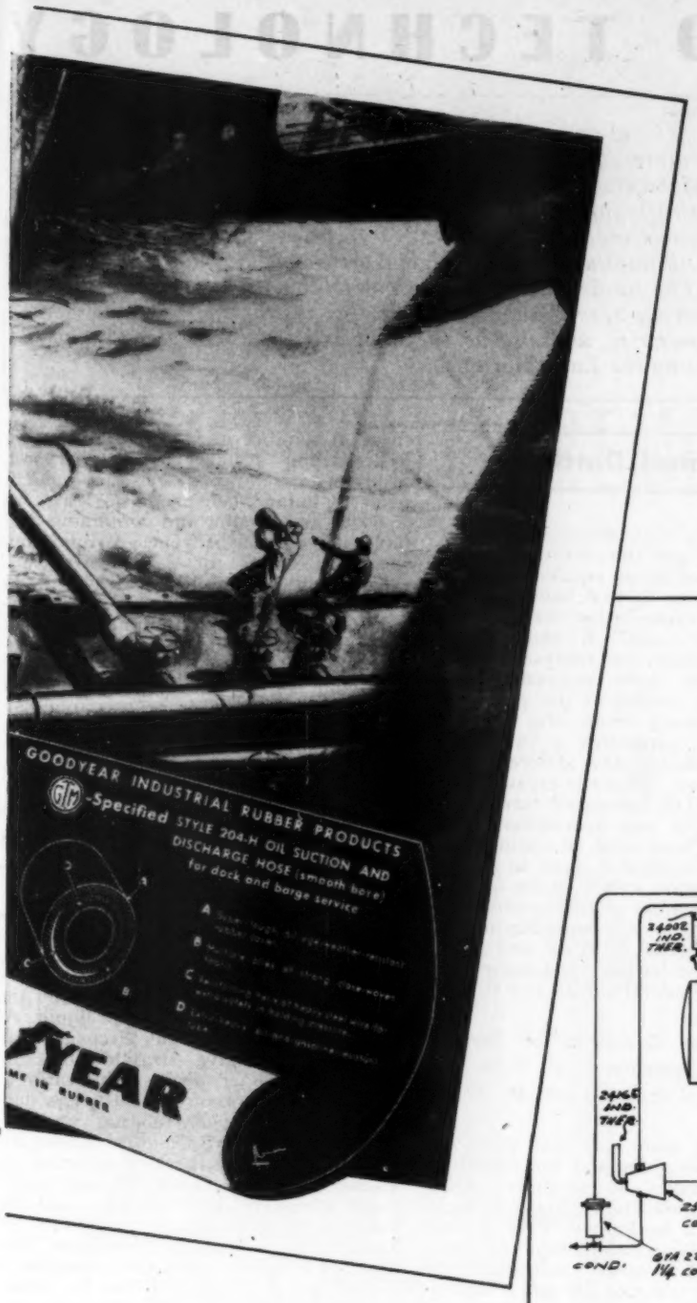
wherein T represents an aryl group and the C atom as shown is attached to other than a hydrogen atom.

**U.S.P. 2,392,853. Refining of Petroleum Hydrocarbon Waxes.** A. Kinsel to L. Sonneborn Sons, Inc.

Corrosive substances induced by treating residual wax with $AlCl_3$ are removed by reacting the wax with alkali or alkaline earth metal plumbites.

**U.S.P. 2,393,335. Reclaiming Used Lubricating Oils.** J. M. Musselman to Standard Oil Company of Ohio.

Used lubricating oil is purified by heating it with a phosphorus sulfide to at least 225° F. and separating precipitated matter.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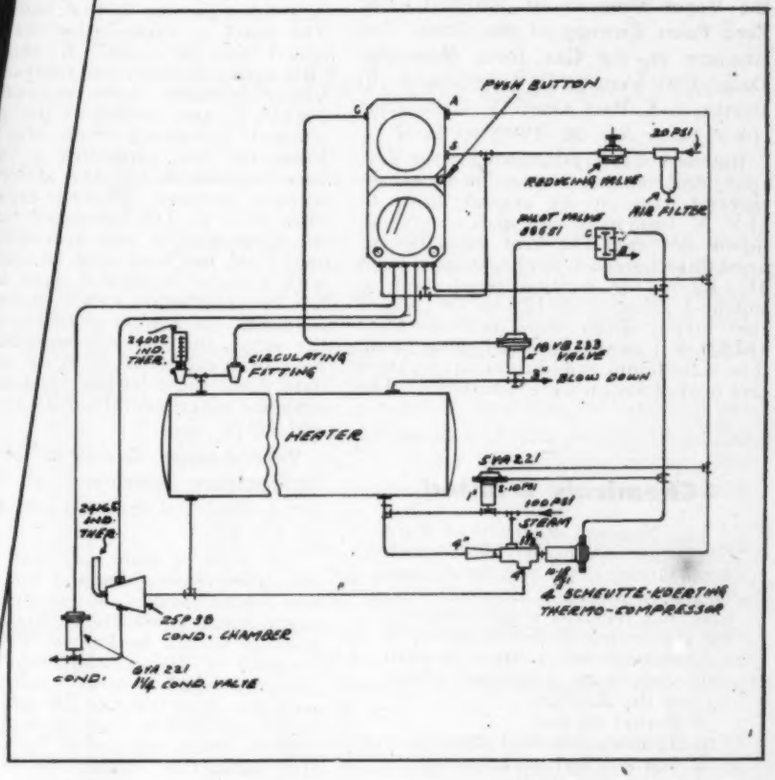
● *This Goodyear Ad appeared in the December 3rd issue of Time Magazine. The oil hose it talks about was vulcanized by a process developed jointly by Goodyear and Taylor Engineers.*




chemicals to high octane gasoline. Not to speak of the gaseous diffusion process at Oak Ridge for which Taylor is prime contractor for process control instruments.

• • •

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Traver Road, Ann Arbor, Michigan
*under supervision of*

DR. E. H. LESLIE and DR. H. B. COATS

*The abstracts here presented are selected from the current literature of science and technology to afford reference to fundamental information not easily available to all readers. Abstracts of articles appearing in readily obtainable trade journals are not included.*

*Photostat copies of original articles will be supplied at cost by The Leslie Laboratories. Complete or limited bibliographies covering special topics by title, by abstracts, or in complete manuscript, will also be prepared and furnished at reasonable cost by the Laboratories.*

## Fundamental Physical and Chemical Data

**The Heat Capacity and Entropy, Heats of Fusion and Vaporization and the Vapor Pressure of Butene-1. The Zero Point Entropy of the Glass. The Entropy of the Gas from Molecular Data.** J. G. ASTON, H. L. FINK, A. B. BESTUL, E. L. PACE AND G. J. SZASZ. *Jour. Amer. Chem. Soc.* **68** (1946) pp. 52-57.

Butene-1 was crystallized for the first time, thus making it possible to obtain thermal data on the crystal down to 11.5° K. leading to entropies of crystal, liquid and gas. The heat capacities of crystalline butene-1 were measured from 11° K. to the melting point, and of butene-1 glass, from 12.5 to 78° K.; of the liquid, from the melting point (87.82° K.) to the normal boiling point. The solid-liquid equilibrium temperature and heat of fusion were determined. The vapor pressures were determined from 199 to 267° K. and the results are seen to be represented by an equation leading to a value for the normal boiling point. The heats of vaporization were determined from 202 to 267° K. and a quadratic equation derived to represent them. Vapor densities were measured at 298.16° K. and a value of the virial coefficient calculated from the results. From the heat capacities, a value has been calculated for the difference in entropy between butene-1 crystal and glass at 0° K. The calculated entropy of the ideal gas at one atmosphere and 266.91° K. has been used, in conjunction with assumed molecular data, to calculate two alternative values of the barrier hindering the mutual relative rotation of the ethyl and vinyl groups. Entropies have been calculated from the molecular data using these barriers and compared with the experimental values at 200, 230, and 298.16° K.

**Vapor-Liquid Equilibria of Binary Hydrocarbon Systems.** J. M. HARRISON AND L. BERG. *Ind. & Eng. Chem.* **38** (1946) pp. 117-20.

The authors point out that the constantly increasing demand for pure hydrocarbons makes it necessary that vapor-liquid equilibrium data for such systems should be known. Present-day methods of design calculation for handling nonideal systems indicate that, with the determination of more vapor-liquid equilibrium data, it will be possible to design equipment for azeotropic and extractive distillation operations with as much precision as is now possible for the distillation of regular mixtures. A number of close-boiling binary hydrocarbon systems were studied. Minimum azeotropes exist for 2,2,3-trimethylbutane-benzene, boiling at 75.6° C. at 736 mm. and containing 56.7 mole percent of benzene. A minimum azeotrope exists for 2,2,3-trimethylbutane-cyclohexane boiling at 79.45° C. at 744 mm. and containing 47.8 mole percent of 2,2,3-trimethylbutane. A minimum azeotrope exists between benzene-cyclohexene boiling at 78.9° C. at 740 mm. and containing 65.7 mole percent of benzene. No azeotrope was found in the systems 2,2,4-trimethylpentane-methylcyclohexane and cyclohexane-cyclohexene.

**Correlating Viscosities of Gases with Temperature and Pressure.** D. F. OTHMER AND S. JOSEFOWITZ. *Ind. & Eng. Chem.* **38** (1946) pp. 111-16.

At constant pressure, the viscosities of gases and of gaseous mixtures can be plotted directly to give straight lines on logarithmic paper when a temperature scale is used that is calibrated from the vapor pressure of a reference liquid. At constant temperature, gas viscosities can be plotted to give straight lines on logarithmic paper against the ratio of the kinematic pressure and the gas density. Methods of correlating gas viscosities, temperatures, and presusres are expanded to allow the estimation of the viscosity of gases under different conditions where their PVT data and the viscosity at one set of conditions are known. A nomograph has been constructed to allow ready determination of viscosities; it is believed that the values so determined fall well within the precision of published experimental data. Thermodynamic derivations indicate the theoretical basis for these plots and the resulting nomograph.

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- Epihydrin aldehyde
- Ethyl peroxy isopropyl carbonate
- 2-Hydroxy-6-chlorotoluene
- β-Hydroxy-α-methyl adipic acid
- Methyl phenyl tin dichloride
- 1,1,2,2-Tetraphenyl ethane
- Dimethyl cyclohexanes
- Trimethyl cyclopentanes
- Methyl ethyl cyclopentanes
- Methyl ethyl hexanes

## Chemical Composition and Reactions

**Catalytic Conversion of Hydrocarbons.** F. E. FISHER, H. C. WATTS, G. E. HARRIS, AND C. M. HOLLENBECK. *Ind. & Eng. Chem.* **38** (1946) pp. 61-64.

In the catalytic conversion of aliphatic hydrocarbons to aromatics, considerable effort has been focused on the oxides of the metals of Group VI of the periodic table as catalysts. Chromium and molybdenum oxides have received special attention. The present paper deals with the activity improvement or promoting effect of antimony tetroxide upon a chromium sesquioxide catalyst. The catalyst used was made by depositing chromium trioxide and antimony tetroxide on an activated alumina carrier instead of coprecipitating the three oxides as a gel, as was done by Burk and Hughes. The activity of the catalyst in the catalytic aromatization of aliphatic hydrocarbons was improved to an appreciable extent. The reaction was carried out using three different types of charge stocks: (1) a heptane fraction from a natural gasoline, (2) a heavy straight-run gasoline, and (3) a thermally cracked gasoline. Both the liquid and gaseous products were analyzed. The production of aromatics during any one reaction period increased through a

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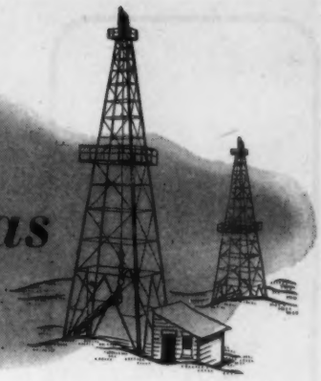

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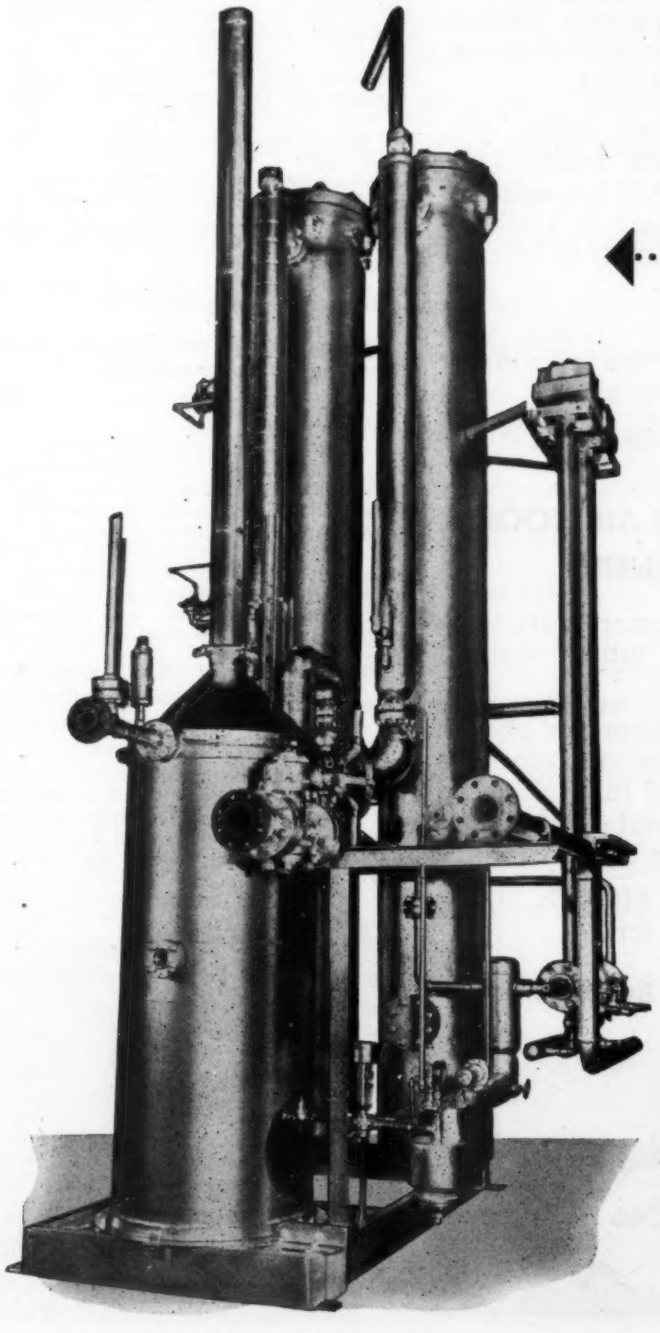

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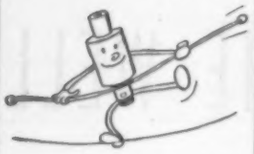

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maximum and then decreased. The yield of gaseous products paralleled the formation of aromatics until excessive cracking suspended the dehydrogenation-cyclization reaction. The yield of toluene from h-heptane was 25.7 percent of the theoretical. Cracked gasoline yielded a liquid product containing 49.5 percent aromatic hydrocarbons from which toluene, xylenes, ethylbenzene, mesitylene, 1,3-dimethyl-4-ethylbenzene, and naphthalene were isolated.

**Polymerization. I. The Reaction of Organosodium Compounds with Butadiene.** A. A. Morton, G. H. Patterson, J. J. Donovan and E. L. Little. *Jour. Amer. Chem. Soc.* **68** (1946) pp. 93-96.

Under the conditions used in the work. butadiene adds to amyl-, benzyl-, and cyclohexyl-sodium. The sodium compounds are decomposed by carbon dioxide or by water and alcohol to give acids or hydrocarbons. The acids and derived esters having saponification equivalents and double bonds that correspond to addition of one or two butadiene units can be isolated readily. Those which have three or more units show unsaturation lower than expected and an oxygen content that is abnormally high. Other properties of the esters suggest that oxygen is absorbed readily. These compounds, whether as esters or the corresponding hydrocarbons, undergo thickening by a thermal polymerization at 230° C. The residues and rubber-like products undergo a similar thermal polymerization.

**The Hydrolysis Rates of the Three Butadiene Monochlorohydrins.** R. G. Kadesch. *Jour. Amer. Chem. Soc.* **68** (1946) pp. 46-49.

The rate constants for the hydrolysis of three butadiene monochlorohydrins by water and by base have been determined. The conversion of the two butadienes a-chlorohydrins to the epoxide by base is extremely rapid. Their second order rate constants have been compared with those of ethylene and propylene chlorohydrins and are discussed with regard to the mechanism involved.

**The Chemistry of Dicyclopentadiene. III. Addition of Alcohols and Phenols.** H. A. Bruson and T. W. Riener. *Jour. Amer. Chem. Soc.* **68** (1946) pp. 8-10.

Dicyclopentadiene forms addition compounds with alcohol and phenols in the presence of acidic catalysts such as $BF_3$ or sulfuric acid to yield rearranged unsaturated ethers, which are derivatives of hydroxy-dihydro-exo-dicyclopentadiene. The lower aliphatic ethers thus obtained possess intense floral odors.

**New Catalysts for Friedel-Crafts Type Reactions.** A. N. Sachanen and P. D. Caesar. *Ind. & Eng. Chem.* **38** (1946) pp. 43-45.

Various typical reactions, such as can be catalyzed by Friedel-Crafts catalysts or strong acids, can be effected in the presence of heterogeneous catalysts of the silica-alumina type or homogeneous catalysts such as the hydrogen halides or organic halides. Silica gel, notwithstanding its enormous surface, does not catalyze the reactions described in the paper. However, a small proportion of the alumina, of the order of 1 percent by weight of the silica, is sufficient to

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produce an active catalyst. Oxides such as thoria or zirconia can be substituted for the alumina. The alkylation of aromatic hydrocarbons with olefins, long established as a Friedel-Crafts synthesis, was the first application of silica-alumina to condensations of this type. Destructive alkylation reactions catalyzed by aluminum chloride have been effected over silica-alumina catalysts. The paper describes the syntheses of anthraquinone and benzophenone as new examples of the application of silica-alumina catalysts to conventional reactions of the Friedel-Crafts type. A bibliography of 24 references is included.

**Determination of Helium Group Gases in Natural Gases and in the Atmosphere.** G. H. CADY AND H. P. CADY. *Ind. & Eng. Chem., Anal. Ed.* **17** (1945) pp. 760-66.

The paper describes a continuation of a study extending from 1905 to the present time. Methods for determining helium plus neon, argon, and krypton plus xenon by procedures involving fractional distillation, fractional desorption from activated charcoal, and absorption of reactive gases by hot calcium are described. The analysis of the atmosphere by these techniques agrees well with data of earlier investigators and indicates that 100 ml. of dry air, free from carbon dioxide, contain $0.0023_3$ ml. of helium plus neon, $0.00012_6$ ml. of krypton plus xenon, and $0.93_8$ ml. of argon. Several natural gases, rich in nitrogen, were analyzed, but none was found to contain a higher proportion of any rare gas, except helium, than is present in air. The subject is reviewed bibliographically, and the apparatus and methods used in the work are described in considerable detail.

**The Mechanism of Paraffin Isomerization.** H. S. BLOCH, H. PINES, AND L. SCHMERLING. *Jour. Amer. Chem. Soc.* **68** (1946) p. 153.

Paraffin hydrocarbons do not undergo isomerization in the presence of aluminum halide catalysts unless traces of olefins or their equivalent are present. This suggests that the isomerization may proceed by a chain mechanism similar to that proposed for the alkylation of isoparaffins with olefins. The suggested reaction mechanism is shown in structural form. Dehydrogenation of the paraffin by reaction with hydrogen chloride or hydrogen aluminum tetrachloride offers another means of obtaining the necessary chain-initiating ion. Experimental evidence in support of this reaction can be found in the observation that small quantities of hydrogen are actually formed during the initial stages of butane isomerization with a supported alumnium chloride catalyst and hydrogen chloride. The mechanism proposed is also applicable to the isomerization of the cycloparaffins.

## Manufacture: Processes and Plant

**Pure Hydrocarbons from Petroleum.** J. GRISWOLD, D. ANDRES, C. F. VAN BERG, AND J. E. KASCH. *Ind. & Eng. Chem.* **38** (1946) pp. 65-70.

Extractive distillation, or Distex process is described in its application to the separation of straight-run fractions consisting of paraffins, naphthenes, and aromatics. Details are given of the apparatus and procedures used to resolve the narrow-boiling hexane fraction. This type of operation has been used in toluene purification, and in the purification of butadiene. The operation involves fractional distillation in the presence of a selective agent or solvent, and is conducted at a temperature at which liquid and vapor phases coexist. The foreign component or agent can be any of several hundred organic compounds, including all of the "selective solvents" used in solvent refining. As a matter of economical operation and ease of separating the solvent from the hydrocarbon extract and raffinate, the solvent should have a considerably higher boiling point than the hydrocarbons. The principal difference between azeotropic distillation and extractive distillation is that in the former a solvent must be selected, the boiling point of which is close to that of the material to be separated. Extractive distillation thus has one more degree of freedom than azeotropic distillation, since the phase compositions are not fixed by either temperature or pressure alone. This permits the solvent in the liquid reflux to be maintained at a desired optimum concentration. The apparatus and procedure used in the work are described in considerable detail, and a typical flowsheet of a commercial method for the separation of pure hydrocarbons from a $C_6$ fraction is given.

**Heat Transfer to Liquids Boiling Under Pressure.** M. T. CICHELLI AND C. F. BONILLA. *Trans. Amer. Inst. Chem. Engrs.* **41** (1945) pp. 755-87.

The temperature difference required to cause boiling from a horizontal chromium-plated surface at medium and high heat transfer rates, and at pressures ranging from atmospheric to near the critical, was measured for water, ethanol, benzene, propane, n-pentane, n-heptane, 50 mole percent water-ethanol, and 33 and 67 mole percent propane-pentane. The coefficient of heat transfer was found to continue to rise as pressure increased, until nucleate boiling ceased to be stable near the critical pressure. An approximate graphical correlation was found for predicting the maximum boiling rate possible at any pressure, and another for the temperature difference required to produce the maximum rate. The maximum rate is found to occur at about one third of the critical pressure. Binary mixtures were found to require several times as large a temperature difference to boil at the same rate as the pure components.

**Effect of Added Components on the Relative Volatility of Two Binary Mixtures.** O. L. UPDIKE, JR., W. M. LANGDON, AND D. B. KEYES. *Trans. Amer. Inst. Chem. Engrs.* **41** (1945) pp. 717-36.

In hydrocarbon systems comprising an aromatic and a naphthene of close boiling points, exemplified by benzene-

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cyclohexane and toluene-methylcyclohexane, the relative volatility is increased by addition of polar organic compounds with high dielectric constant; two substances (acetonitrile and nitromethane) with dielectric constants of about 39 have especially high improvement factors, while substances with dielectric constants above 40 have especially low improvement factors. Three substances (acetonitrile, furfural, and nitromethane) with dipole moments between 3.5 and $3.8 \times 10^{-18}$ e.s.u. have especially high improvement factors. At lease one substance (nitromethane) with a high improvement factor has an internal pressure of about 5000 atmospheres. However, the improvement factor does not appear to be at all well defined by the variables of dielectric constant, dipole moment, and internal pressure. Highly polar compounds like water and formamide gave very little improvement, because of their limited miscibility with the hydrocarbons.

**Film Resistances in Rectification.** R. E. PECK AND E. F. WAGNER. *Trans. Amer. Inst. Chem. Engrs.* **41** (1945) pp. 737-53.

A method was developed for determining the relative film resistances in rectification. It was applied to the experimental results obtained by the authors and to data in the literature. Study of the systems methanol-water, acetone-water, isopropanol-water, and ethanol-water in a wetted-wall tower showed that approximately 100 percent of the total resistance to diffusion was in the gas film. However, in plate columns and fiber-glass-packed columns the specific resistances in the two phases are about equal. Knowledge of the relative gas- and liquid-film resistances permits more accurate design of rectifying columns by the H. T. U. method. For improvement in practical column design, attention must be directed toward decreasing both film resistances.

**Limiting Capacity of Dumped Tower Packings.** W. E. LOBO, L. FRIEND, F. HASHMALL, AND F. ZENZ. *Trans. Amer. Inst. Chem. Engrs.* **41** (1945) pp. 693-710.

It was found that the use of the correct values of surface and free voids is necessary to obtain a good correlation of flooding velocity in packed columns. The characteristics of several packings were investigated and results are given in tabular form. A correlation of published data on flooding velocity in dumped packed towers is presented that predicts the flooding velocity within 11.5 percent. Groups of variables used were the same as those used by Sherwood, Shipley, and Holloway. However, experimentally determined values of $a/F^3$ varied considerably from previously published or reported values.

**Flow of Liquids through Vertical Circular Orifices and Triangular Weirs.** F. W. GREVE. Purdue Univ. Eng. Expt. Sta., Res. Ser. No. 95 (1945) 67 pp.

Coefficients of discharge for vertical sharp-edged orifices from diameters of 0.252 to 2.00 inches were determined using cylinder oil, furnace oil, mixtures of these oils, soap solutions, sugar solutions, and water. All liquids discharged into air from a tank with adjustable head. In every instance the coefficient increased at first with increase in head, then reached a maximum or critical


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value, and then decreased. The critical discharge coefficient varies inversely with diameter of orifice, but is independent of liquid and temperature. The critical coefficient occurs at a critical head that depends on the diameter of the orifice and on the kinematic viscosity of the liquid, and is a minimum at a viscosity of 0.00003 ft.²/sec. There was no correlation of the coefficient with either Reynolds or Weber number. A free jet of water would spring clear from a ⅛-inch orifice when the ratio of head to diameter was 5.6. However, an SAE 140 gear lubricant did not produce a free jet even when the ratio was 488. Discharge coefficients were also determined for 30, 60, and 90° weirs for water and for mixtures of alkanol and water. The coefficient decreased as the head increased. Surface tension and Weber number did not affect the coefficient.

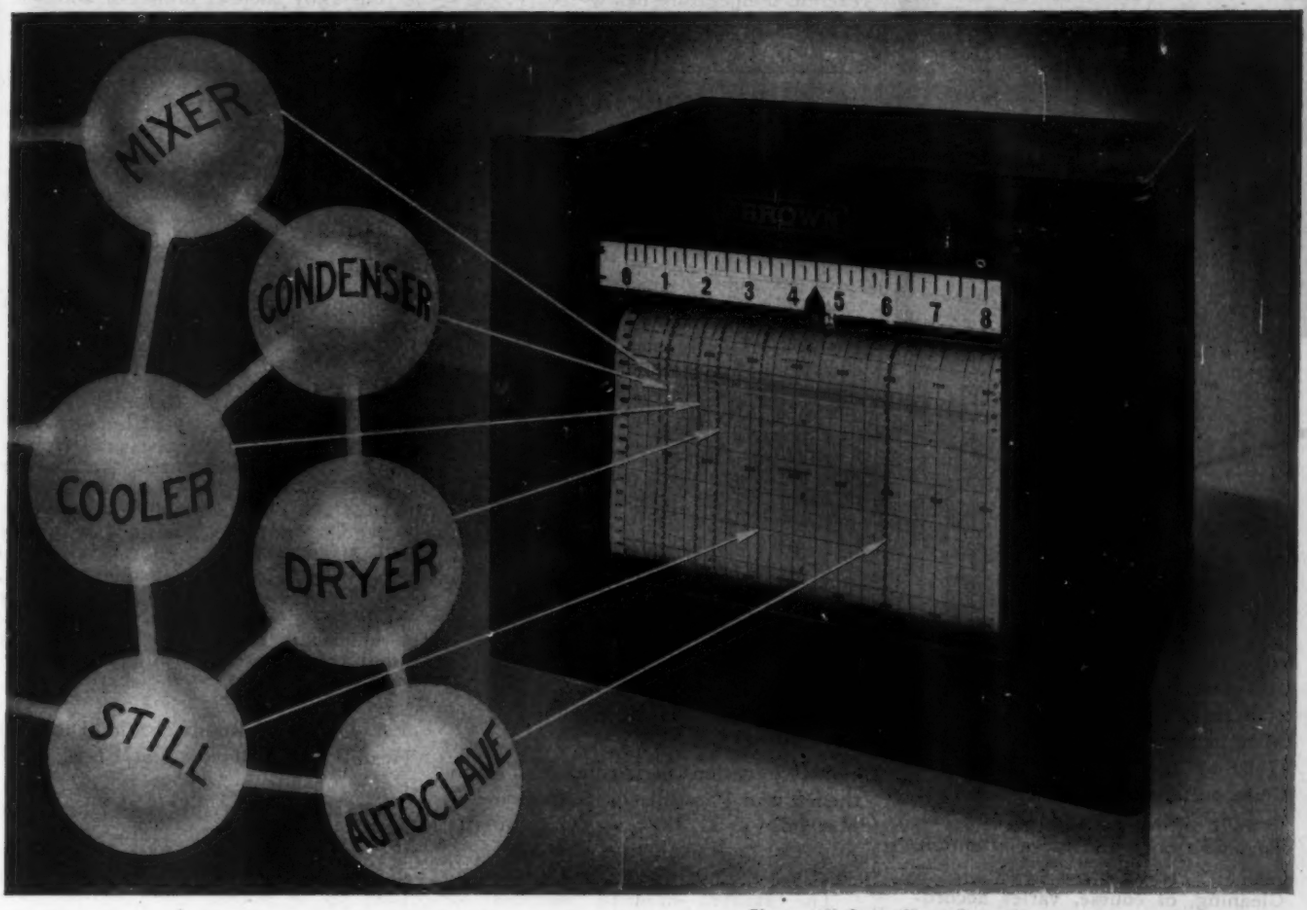
**Extractive Distillation of Hydrocarbon Mixtures in a Packed Column.** R. S. Dicks and C. S. Carlson. *Trans. Amer. Inst. Chem. Engrs.* **41** (1945) pp. 789-803.

The authors give a comparison of the separations obtained in the same apparatus under conditions of extractive distillation and fractional distillation. n-Heptane-methylcyclohexane was used as a test mixture. The separation, effected by extractive distillation using aniline as the auxiliary agent was equivalent to a fractional distillation tower four times as tall as the one actually used. The constant-boiling mixture cyclohexane-benzene was separated by extractive distillation yielding pure cyclohexane and benzene. Methylcyclohexane-toluene solutions were also nearly completely separated. The design of a batch countercurrent apparatus suitable for extractive distillation is described.

**Experiences in the Commercial Application of the Hydrogen Fluoride Alkylation Process.** E. M. Skinner. *Trans. Amer. Inst. Chem. Engrs.* **41** (1945) pp. 647-70.

The paper describes a commercial hydrogen fluoride alkylation process plant, and prevents the operating data and maintenance experiences that were gathered over a long period. The feed stock, comprising butanes and butylenes, is intimately mixed with substantially anhydrous HF acid to effect the alkylation of isobutane with butylenes. The mixture of hydrocarbons and acid catalyst is settled continuously. The acid phase is returned to the mixing zone while the hydrocarbons flow on through various stages of distillation for purification and separation of the several products. The process is flexible, and the operating variables are not critical as regards optimum performance. Yield and quality of the product is controlled to a large extent by the isobutane-olefin ratio in the feed to the reaction stage. The optimum ratio is approximately 10:1. The continuity of the operation depends upon the condition of the acid catalyst. A direct titration of the acidity of the catalyst does not give an adequate definition of its usefulness. If the water content and the content of higher-boiling acid-soluble oil in the acid exceeds 10 percent, excessive quantities of hydrogen fluoride are carried from the reaction zone in the form of organic

# CONTROL OF TEMPERATURE . . .

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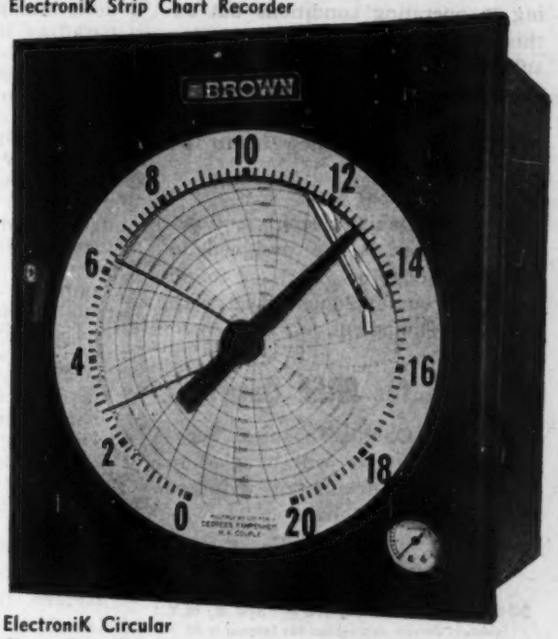


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fluoride. The percent of acid in the reaction stage emulsion should be maintained above 40 percent and improved results are obtained by operating at a reaction temperature below 100° F. Carbon steel is satisfactory as a construction material for all stationary equipment and for machinery parts that have large clearances. Moving parts of close clearances should be fabricated of a nonferrous alloy such as Monel or alloys of high copper content.

## Products: Properties and Utilization

**Determination of Water Content in Oils.** M. M. Acker and H. A. Frediani. *Ind. & Eng. Chem., Anal. Ed.* **17** (1945 pp. 793-94.

Recent publications have indicated that the Karl Fischer reagent has great merit in the determination of water. However, pitfalls may be encountered in adapting this reagent and technique to a new control method. Properly employed, with due regard to necessary precautions in manipulation, the reagent lends itself to the accurate and rapid determination of water. Improperly used, however, it may give erroneous results and lack of reproducibility. The adaptation of the method to the determination of moisture in insulating and lubricating oils, either new or used, is possible only with careful and painstaking adherence to a predetermined uniform technique. Reagents, apparatus and procedure recommended are described in considerable detail. Tabular data are given for determination of a light oil and on a transformer oil.

**Determination of Aromatics, Naphthenes, and Paraffins by Refractometric Methods.** R. M. Gooding, N. G. Adams, and H. T. Rall. *Ind. & Eng. Chem., Anal. Ed.* **18** (1946) pp. 2-13.

The authors describe methods for determining aromatics, naphthenes, and paraffins in virgin naphthas and condensates boiling below 320° F. Up to 243° F. most of the individual hydrocarbons can be estimated. Efficient fractionation is used to produce narrow-boiling-range fractions containing relatively few individual hydrocarbons. Densities and refractive indices for the mercury g line and the sodium D line, determined for each fraction at 20° C., give specific dispersions and refractivity intercepts used for analysis of the samples. Aromatics were determined from specific dispersions and naphthenes, and paraffins were determined by the use of modified Kurtz and Headington refractivity intercept-density charts. The aromatics can be determined from these charts with some loss in accuracy. Development of the charts from experimental and literature data is discussed and illustrated in some detail. Analyses of 10 crude-oil naphthas are given.

**Determination of 4-Vinyl-1-cyclohexene (Butadiene Dimer) in Recycle Styrene.** H. A. Laitinen, A. S. O'Brien, and S. Wawzonek. *Ind. & Eng. Chem., Anal. Ed.* **17** (1945) pp. 769-72.

The determination of butadiene dimer in recycle styrene is of importance not only in determining the purity of the recycle material but also in allowing more accurate material balances in the polymerization process. Butadiene dimer was determined in recycle styrene by quantitatively polymerizing the styrene, using sodium as a catalyst. The vinylcyclohexene is determined by the iodine chloride method. The method is sensitive to 0.2 percent vinylcyclohexene and is accurate to about ± 0.2 percent (absolute) over a range of 1 to 20 percent vinylcyclohexene.

**Improved Device for Decomposition of Grease.** R. W. Tarara. *Ind. & Eng. Chem., Anal. Ed.* **18** (1946) p. 68.

The author used a method for the decomposition of grease that cuts the time of decomposition from 20 minutes to 5 minutes for a light grease, and from 2 hours to 20 minutes for the heavier grades. The method used was an adaptation of the so-called boiling method in which 30 grams of grease were placed in a 400-ml. beaker, and 200 ml. of 10 percent hydrochloric acid solution added, and the mixture heated to the boiling point. An inverted funnel was used as a percolator to release vapor and prevent bumping, and at the same time to accomplish stirring.

**A Constant Reflux Ratio Distilling Head.** L. Berg. *Ind. & Eng. Chem., Anal. Ed.* **18** (1946) pp. 54-5.

The author describes a distilling head that gives a constant reflux ratio regardless of distillation rate, that has no moving parts, and that can be readily built by a skilled glass blower. The apparatus is of relatively low holdup and of low cost of construction. It can be used under vacuum or under pressure, and can handle heterogeneous as well as homogeneous condensates.

**Improved Distilling Head for Laboratory Fractionating Columns.** B. G. Zimmerman. *Ind. & Eng. Chem., Anal. Ed.* **17** (1945) p. 815.

The author describes a glass still head of the total condensation type. It permits determination of the reflux ratio. Once this ratio is set, it will remain constant. Temperatures of the reflux products are easily taken. The head can be operated under total reflux. It has a low holdup. The head can be made to fit any size fractionating column for laboratory or pilot-plant use.

**Glass Perforated Plate Distillation Column.** W. M. Langdon and D. J. Tobin. *Ind. & Eng. Chem., Anal Ed.* **17** (1945) pp. 801-5.

Plate columns have been preferred for many fractional distillation operations in the laboratory because of large throughput and relatively constant operating characteristics. However, except in the case of the metal sieve plate columns, the difficulties involved in their construction have resulted in the general use of packed or film-type of laboratory columns. The column described by the authors employs perforated glass plates that are easily constructed. The plates, downpipes, and plate spacers can be inserted into the column without sealing.

# NEW EQUIPMENT

## . . . MANUFACTURERS' · LITERATURE . . .

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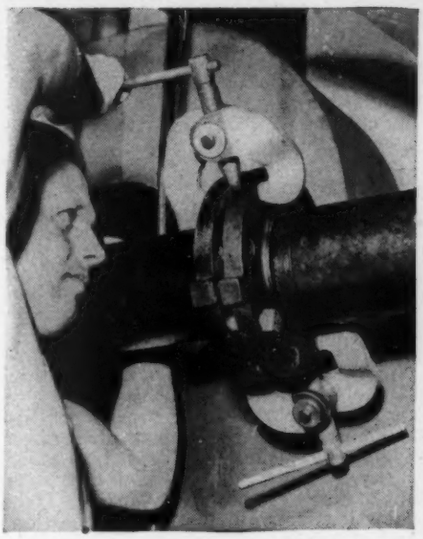
### 1—Tool For Replacing Pipe Gaskets

T. G. Persson Company, 224 Glenwood Avenue, Bloomfield, New Jersey, has announced a tool which opens pipe flanges for gasket renewal. With "Flange-Jacks," pipe flanges may be opened or closed without damage to flange faces, and without danger from sparks in the presence of inflammable vapors or danger from flying wedges or chips.

The jacks operate with the minimum of labor and extra pressures smoothly and evenly, eliminating vibration which might cause leaks in nearby joints. They are capable of opening joints against a load of 15 tons. Flanges are opened evenly and bolt holes remain in alignment. Closure is accomplished in a similar manner.

Standard size jacks open 2- to 20-inch flanges. Simple in design, the jaws are one-piece steel forgings, and screw points are case hardened.

CHECK THE CARD AT PAGE 225



Flange. Jacks

### 2—Automatic Control Actuator

Harold Beck Company, 3544 North Second Street, Philadelphia 40, is publicizing the Beck Mechanism, a highly-developed automatic control actuator which operates valves, rheostats, autotransformers and similar devices. It makes close control possible under rapidly changing loads, holds temperature or other conditions to an exact control point free from the drooping characteristic, and permits operation at maximum sensitivity. This triple function control is based on three control actions.

C-1 is proportional to and opposes any change of the controlled condition within the throttling range. If this were the only control action, a return of the condition to the control point would place the regulating device in the position from which it started and, on a sustained load change, continuous hunting would result, or if the throttling range were corrected to avoid hunting the process would settle down at some value off the control point. C-2 and C-3 are the automatic reset or droop corrections which eliminate this effect. The C-2 is the major part of the correction. It is proportional to the C-1 action, and is introduced to the system as fast but not faster than it can be absorbed. The C-3 action consists of an impulse that is periodically introduced when the system is off the control point, to move the regulating device in the proper direction to restore the system to the control point. This is a timed action and is proportional to the magnitude of the deviation. C-3 prevents the system from staying off the control point due to some unusual combination of demand change and control action.

Beck Mechanisms handle special control problems involving sequence action interlocks, directional contacts, maintaining contacts, and other switching arrangements. They are used for control of gas-fired or electric furnaces, automatic reversal of regenerating furnaces, safety circuits for gas purging and in electrical testing apparatus. The solution of many more industrial control problems can be simplified and improved by taking advantage of the many unique features of this control mechanism, the manufacturer anticipates.

Each Beck Mechanism is driven by a slow-speed, high-torque, non-coasting Beck control motor. A non-mechanical magnetic brake assures accurate reproduction of minimum turn down and non-hunting control action without sacrificing sensitivity of the control system. The heavy motor shaft turns in grease-sealed ball bearings. The armature can be stalled without damage to the mechanism and the stalled motor will not overheat.

Detailed engineer information can be obtained by using the card.

CHECK THE CARD AT PAGE 225

Beck Mechanism

### 3—Fire Control Nozzle

Blaw-Knox Sprinkler Division, Pittsburgh, has announced a new fire-control nozzle for fixed installations, constructed in such manner that it produces a fog. Named the "Aquatomic Fog Nozzle," it produces fog by forcing water through three clear spiral passages and a clear central passage in the head of the nozzle. The fog pattern is maintained even at greatly reduced water pressures.

The fog nozzle is well adapted to the petroleum, chemical, and electrical industries.

CHECK THE CARD AT PAGE 225

### 4—Coupling

Roylyn Mechanical Laboratory, 8928 Santa Monica Boulevard, Los Angeles 45, has announced a quick coupling for use on hoses and lines operating under pressure. The new coupling is particularly adapted for hydraulic and pneumatic lines from pressurized tanks, such as truck and trailer connections, fuel lines, paint sprayers and air-driven tools.

Based on the "inclined plane and wheel principle," the coupling employs only three major parts: cam ring, ball cage, and nipple. By rotating the cam collar steel balls are forced inward into a groove in the nipple forcing the halves together. The gasket retained in the ball cage provides a positive seal against dirt as well as being the separating spring force necessary to the operation of the lock. A slight shoulder on the cam assures a positive lock; application of more pressure tightens it, and only the cam collar can unlock it.

Tests on an alloy-steel model showed a temperature operating range from minus 65° F. to 300° F. with safe pressure limits of 1680 psi for the 2-inch

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coupling, and 11,500 psi for the ¼-inch unit.

Couplings are available in aluminum, brass, alloy steel and stainless steel, with gaskets to withstand oils or acids. Pipe threads or flared tube connections are optional, with caps to fit all sizes of nipples. Standard thread sizes run from ¼- to 2-inch, with larger sizes available on special order.

Literature on industrial applications of these and allied products are available.

CHECK THE CARD AT PAGE 225

### 5—Sealing Compound

Parker Appliance Company, Cleveland, Ohio, has announced "Uniseal," a general-purpose thread- and gasket-sealing compound, claiming that it produces a high-efficiency seal which is proof against air, water, steam, gas, gasoline, oil, hydraulic fluids and aromatics. The product is a paste of uniform consistency, containing no free metallic particles which might set up galvanic action, or other additives subject to washing out. It flows smoothly to form ribbon gaskets, and blends readily with cut gasket material. It possesses good anti-seize characteristics which aid in the assembly of threaded fittings. Solubility in alcohol and tetrachloride simplifies any cleanup which might be necessary. Disassembly and rearrangement of work on which the compound is used is made easier as the seal breaks cleanly with application of minimum force. Tests show it effective under severe vibration, and at temperatures above 300° F. It is available in tubes and container from 8 ounces to 5 gallons.

CHECK THE CARD AT PAGE 225

### 6—Feedwater Control

Combustion Control Corporation, 77 Broadway, Cambridge 42, Massachusetts, has announced Fireye Boiler Feedwater Control, Type B177N, designed to provide automatic control for boilers. It maintains desired water level through valve or pump control, and cuts off fuel supply and sounds an alarm at a predetermined low-water level. The instrument operates without any moving mechanical elements: the only contact with the boiler is through an auxiliary fitting containing metal probe rods which determine the control levels. There are no moving elements and no vacuum tubes.

The probe fitting is mounted parallel to the water column and wired to the control. The probes within the probe fitting mark pump-on and off levels and a low-water danger point. When water falls below the middle probe, the instrument closes the circuit controlling the pump, and water feeds into the boiler until the top probe is reached. The lowest probe defines the danger point below which boiler water must not fall. Should water drop below this safety probe, the fuel supply is shut off and an alarm sounds.

Bulletin 103-C gives specifications and installation details.

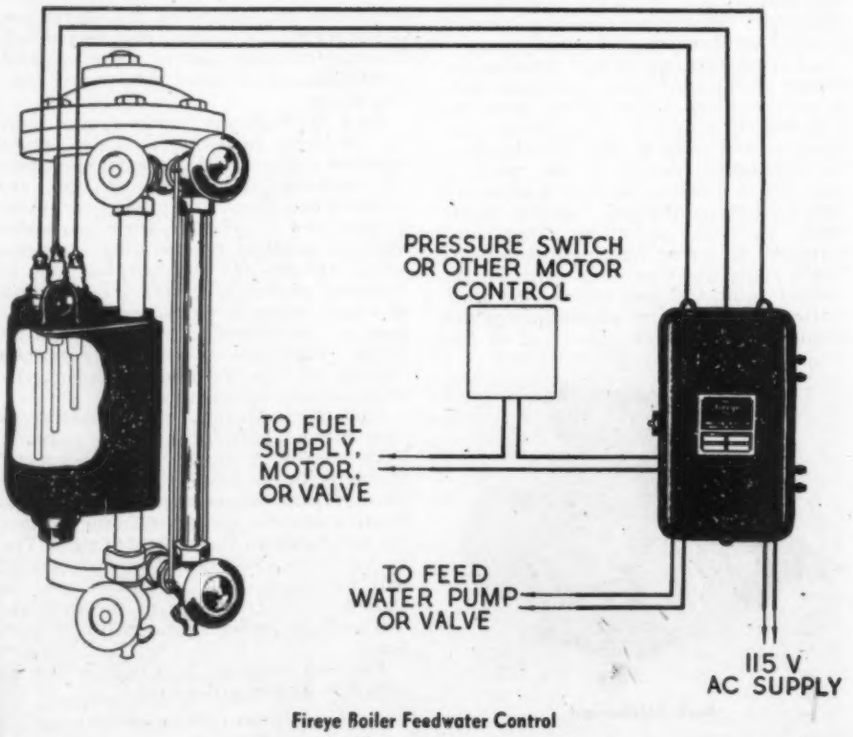
CHECK THE CARD AT PAGE 225

### 7—Operational Recorder

Ess Instrument Company, Bergenfield, New Jersey, has announced availability of its operational recorder to provide a continuous record of "time on" and "time off" of multiple operations, including process timing, periodical operations which can be connected electrically, time study work and the time when a particular check is made, etc.

This instrument can be provided with a single-acting pen, two-position double-acting pen or a three-position pen. As many as six single- or double-acting pens can be combined on a single chart. It can be supplied in 115 to 230 volts a.c. or with special voltage. Clock mechanisms are available from 15-minute periods to 8 days.

The double-acting pen now is being used in Ess "Normality" indicators to record operations. When operation is normal a circle is traced. If the process goes too far in one direction, the pen moves outward and remains there until




Fireye Boiler Feedwater Control

Ess Operational Recorder

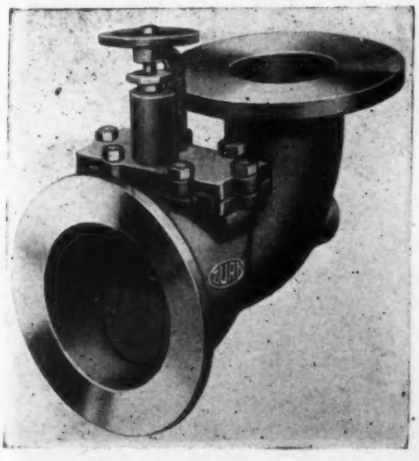
"normal" is resumed, when the pen returns to the center point. If the process goes too far in the other direction the pen moves to the outside of "normal" and remains there as long as the condition persists. As many as six of these three-position pens can be placed on one chart to give a complete picture of the "normality" of six processes by a glance at the chart.

CHECK THE CARD AT PAGE 225

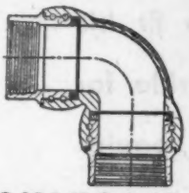
## 8—Discharge Check Valve

J. A. Zurn Manufacturing Company, Erie, Pennsylvania, is in production with a discharge check valve designed to give positive protection against back-surge of discharge lines carrying steam, sludge and other waste liquids in gravity-flow installations.

These units have a swing check flap suspended from a full-floating pin fulcrum to insure positive full-surface contact between ground face of flap and flap seat. The knob in the center of the top surface of the swing check flap contacts the valve-body roof at only one point when the swing check flap is wide open. This prevents the swing check flap from touching the sides of the valve body and assures free operation of the swing check flap. Location of swing check flap in relation to interior of valve body provides ample protection against sediment blocking the valve. Swing



Zurn Discharge Check Valve



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check flap suspended from cover can be removed and replaced.

Discharge check valves may be operated with or without a manual control or with an extension shaft or flexible cable from an access box. The units are hydrostatically tested. Flanges are 150-pound flat-faced, and blank or drilled as specified. They are available with 180-, 105- or 90-degree bodies made of cast bronze, steel, semi-steel, cast iron or alloys for corrosive conditions.

CHECK THE CARD AT PAGE 225

### 9—Hardened and Ground Die Heads

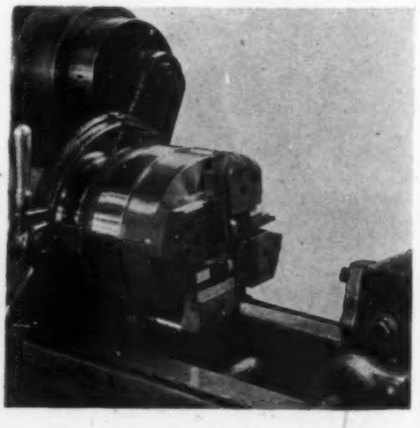
Landis Machine Company, Waynesboro, Pennsylvania, announces availability of a new series of hardened and ground die heads, to supplement its present line. Heretofore, hardened and ground die heads of the "Lanco" type have been available up to the 7/8-inch size. The new series identified as the Type VV are available in the 1-, 1½-, 2- and 2½-inch sizes for use on hand-operated threading machines. These heads are especially recommended for threading alloy steel and for work which must be held to extremely close tolerances.

The Type VV Die Heads have all parts made from special alloy steel, precision ground to assure maximum accuracy.

Chaser holders are clamped to chaser holder slides of heavy cross section to assure maximum rigidity. The manner in which the chaser holders are clamped to the holder slides permits the removal of the holders from the face of the head for interchange without disassembling any part of the head or removing the head from the spindle.

The chaser holder slides are gibbed to the head body to provide compensation for wear. These die heads have remarkably few operating parts, and the inclosed design prevents the entry of chips of dirt to assure maximum life. Zerk type fittings are provided to force heavy grease into the chaser slides and other operating parts to prevent the entry of fine cuttings which would cause excess wear. Extremely fine adjustment for size with the Type VV Head is obtained by means of a graduated adjusting ring at the rear of the head. Size adjustments to the fraction of a thousandth can be obtained.

The Type VV die head employs the new "Lanrac" chaser and chaser holders which was recently announced by Landis Machine Company. The new chaser and



Landis Type VV Die Head